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PERFORMANCE EVALUATION OF FULL-SCALE HAZARDOUS WASTE INCINERATORS

VOLUME V

APPENDICES K AND L

by

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16. ABSTRACT

This report is Volume V, Appendicies K and L, of a series of extensive performance data EPA obtained through eight (8) field tests of actual industrial/commercial incinerators, was prepared in response to the Agency's need to conduct a regulatory impact analysis (RIA) for hazardous waste incinerators to determine the need, adequacy, and economic feasibility of current RCRA incinerator regulations. Tests involved trial burn-type testing at each plant during typical albeit closely monitored daily operations on wastes typical of their routine disposal function.

Most incinerators were found to be closely approaching or performing within RCRA requirements for destruction of compounds, although some facilities showed substandard results in other areas.

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report is one of a five-volume series of reports presenting the recent testing and evaluation results of the performance of eight (8) commercial and industrial hazardous waste incinerators. The Agency's purpose was to gather accurate data on full-scale incinerator environmental performance, for the EPA's use in developing a regulatory impact analysis document in the near future for all such incinerators subject to existing RCRA standards. Because the performance information contained herein is of high interest to the hazardous waste incineration technical community in general, EPA wishes to make it available as a technical report at this time.

The material contained in this report has been reviewed and approved by experts within the Agency with state-of-the-art knowledge of the operation, testing, and evaluation of incinerator performance. During sampling and analysis of the incinerators in the field, EPA's most modern and scientific methods were employed along with the appropriate level of quality assurance and quality control. The incinerators were studied during a closely monitored mode of operation while disposing of mixtures of real-world hazardous wastes typical of their usual feedstocks. We are pleased to report that under these circumstances the incinerators were generally successful in performing their task of safely destroying hazardous waste and controlling other harmful emissions to the environment with some exceptions.

Requests for further information regarding this report on performance of full-scale incineration facilities should be directed to the Energy Pollution Control Division, IERL, Cincinnati.

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ABSTRACT

This report describes a study to evaluate current baseline performance of hazardous waste incinerators. Tests were conducted at eight full scale facilities to characterize all feed and effluent streams. Pollutants measured included EPA Appendix VIII hazardous compounds, particulates, hydrogen chloride, carbon monoxide, and total hydrocarbons. The data were analyzed to address questions on Destruction and Removal Efficiency (DRE), products of incomplete combustion (PIC), pollutant emission levels, performance of air pollution control systems, and relationships of process parameters to destruction efficiency and emission levels. A large base of data on performance of hazardous waste incinerators was accumulated, and many possible relationtionships between parameters were explored. Conclusions included: DREs were generally above 99.99%; measured DREs tended to be higher when concentration of the hazardous compound in the waste was higher; the most frequently observed PICs were benzene, toluene, chloroform, tetrachloroethylene, and naphthalene; the particulate EPA regulation of 180 mg/Nm^3 was not routinely met; and the hydrogen chloride EPA regulation almost always met.

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APPENDIX K

EXTERNAL QUALITY ASSURANCE PROGRAM

SECTION 1.0

INTRODUCTION

The principal technical objective of this EPA program was to conduct sampling and analysis of emissions from hazardous waste incinerators for the purpose of establishing the current baseline of incineration practice. The primary program tasks were sample collection at hazardous waste incinerator facilities and subsequent analysis activities. As a minimum, the sampling at each site required the collection of waste feed composite samples and measurements of the stack effluent to determine destruction removal efficiency (DRE). Other samples, such as combustor ash and scrubber effluent, were collected as appropriate to provide additional information.

Three separate project quality assurance plans were prepared for the sampling and analysis phases of the program to support the QA activities. These plans were for sampling, analysis, and external quality assurance activities and described their respective approaches for quality assurance.

This Appendix presents the results of the external quality assurance activities described in the external quality assurance plan. External monitoring included performance and systems audits of field and laboratory functions.

SECTION 2.0

EXTERNAL AUDIT METHODOLOGY

Performance and systems audits were carried out for both field sampling and laboratory analysis. Performance audits were intended to measure the accuracy and precision of reported numerical values against known values. Systems audits were intended to determine adherence to protocol, procedures, and accepted laboratory practices.

2.1 FIELD AUDIT

The purpose of the field audit was to determine the overall performance of field sampling equipment against standard measurements and to examine adherence to field protocol and procedures. Overall, both audits combined assist in ensuring that the samples are representative. External quality assurance performance and systems audits were conducted at the Ross, Plant B, and Du Pont test plant sites.

2.1.1 Performance Audit

All field performance audits were conducted on site during actual operation of the equipment.

The procedure used for the performance audit is given below.

- Calibrate audit equipment against standard reference devices.
- Requisition manufacturer certified span gases.
- Review EPA standard methods and/or other test protocols.
- Carefully pack equipment for shipment via transportation vehicle.
- Supply span gases (unknowns) to analysts at the test site for quantification.
- Directly compare measurement devices (meter boxes, dry gas meters, RAC console, etc.) against audit device.
- Record all data on performance audit forms. In general, all reported values should be within ±10% as compared to the audit device.
- Prepare and submit a summary report to the crew chief and QAM.

The devices audited and the audit devices are shown below:

Device to be audited	Audit device
Meter box, modified Method 5	Critical orifice (average of 3 readings)
Barometer	Calibrated barometer
Horiba NDIR	Scott standard gas mixture (CO_2)
Beckman NDIR	Scott standard gas mixture (CO)
Beckman 402	Scott standard gas mixture (THC)
Orsat	Scott standard gas mixture (O_2 and CO_2)
Polarigraph 7003	Scott standard gas mixture (O_2)
RAC console temperature gauge	West Pyrotest QB thermocouple
Dry gas meter	Calibrated rotometer

2.1.2 Systems Audit

The intent of the systems audit was to look at all aspects of equipment configuration, data recording, protocol availability, personnel qualification, equipment calibration and maintenance, etc.

A field systems audit form was prepared prior to departure. Procedural aspects during the three phases of sampling (pre-sampling, sampling, and post-sampling) were examined. The auditor actually witnessed the three phases and checked the appropriate column "yes," "no," or "N/A" (not applicable). Comments were entered as necessary.

The systems audit procedure which was used is given below:

- Determine which tests are to be performed.
- Review test procedures and protocols.
- Monitor pre-test preparation.
- Obtain standard audit form.
- Observe the performance of each task.
- Ask questions as required.
- Take corrective actions as necessary.
- Fill in appropriate blank lines on audit form.
- Prepare and submit summary report of findings to crew chief and QAM.

The areas to be audited and the audit mechanisms are shown below.

Area to be audited	Audit mechanism
Modified Method 5 train	Standard audit form
Volatile organic sampling train (VOST)	Standard audit form
EPA Standard Methods 1, 2, 3 and 4	Standard audit form
Process data	Standard audit form
Scrubber effluent grab sample	Standard audit form
Bottom ash grab sample	Standard audit form
Waste feed grab samples (drums, organic wastes, aqueous feed, solids)	Standard audit form
Continuous monitors (Beckman 402, NDIR, etc.)	Standard audit form
Orsat	Standard audit form
Integrated bag samples	Standard audit form

2.2 LABORATORY AUDIT

The main purpose of the laboratory audit was to examine analytical performance and procedural aspects of the analytical laboratory work.

2.2.1 Performance Audit

Performance audit samples were prepared for POHCs, metals analysis, and chloride analysis.

To be audited	Audit material
Volatile organic analysis by GC/MS	Selected volatile organics in methanol
Semivolatile organic analysis by GC/MS	Selected semivolatile organics in dichloromethane
Trace metals analysis by ICP	Selected metals in Ultrex® nitric acid/Milli-Q® water
Chloride analysis by Technicon Autoanalyzer®	Chloride in Milli-Q® water

The procedure used to prepare the samples is given below.

- Selected POHCs (volatile and semivolatile)
  - Select compounds and concentration range so as to approximate actual sample concentration.
  - Obtain organic stock solution.
  - Dilute stock solutions with methanol (volatiles) or dichloromethane (semivolatiles) to 5 to 10X the detection limit or to approximately the actual sample concentration.
  - Record volumes, calculations, concentrations, etc., in record book.

  - Simultaneously assign a performance audit sample number to each sample.
  - Distribute samples to analytical coordinator.

- Metals
  - Select metals and concentration range so as to approximate the actual sample concentration.
  - Obtain stock solution.
  - Dilute stock solution to 5 to 10X detection limit with Ultrex® nitric acid and Milli-Q water.
  - Record volumes, concentrations, calculations, etc., in record book.
  - Simultaneously assign a performance audit sample number to each sample.
  - Submit audit sample to analyst.

- Chloride
  - Select concentration range so as to approximate the actual sample concentration.
  - Prepare stock solutions from which audit samples were prepared.
  - Record weighings, dilutions, and calculations in record book.
  - Simultaneously assign a performance audit number to each sample.
  - Submit audit sample to analyst.

2.2.2 Systems Audit

The purpose of the laboratory systems audit was the examination of the procedural aspects of laboratory operations as opposed to the determination of actual numerical values by analysis.

The systems audit procedure which was used is given below.

- Select the area or procedural item to be audited.
- Review "Program and Quality Assurance Planning Document for Analytical Support of Sampling of Hazardous Waste Combustion Device" by G. A. Jungclaus.
- Obtain audit form.
- Observe conduct of work and ask questions as necessary.

- Take corrective actions as necessary.
- Fill in form as appropriate.
- File summary report.

The systems audit evaluated the following:

- Sample log-in.
- Sample inventory control.
- Sample custody document control.
- Sorbent preparation.
- Glassware preparation.
- Waste sample homogenization and aliquotting.
- Sample spiking.
- Extraction of XAD-2, particulate filter/probe rinse, and condensate water.
- Waste feed extraction for volatiles analysis.
- Waste feed extraction for semivolatiles analysis.
- Methylene chloride extract drying and concentrating.
- Sample digestion and leaching for metals analysis.
- Mass spectrometer calibration.
- GC/MS analysis of volatiles.
- GC/MS analysis of semivolatiles.
- ICP calibration.
- ICP analysis of metals.
- Data handling.

SECTION 3.0

EXTERNAL AUDIT RESULTS

3.1 FIELD AUDIT

3.1.1 Performance Audit

Tables K-1 to K-3 summarize the results of the field performance audit for the Ross, Plant B, and Du Pont plants. These data indicate that the sampling devices which were audited were performing in an acceptable manner. The relative percent error (% E) was calculated as follows:

$$\% E = \frac{RV - TV}{TV} \times 100$$

where RV = reported value
TV = true value

When RV = TV, then % E = 0, indicating no deviation from the true answer.

3.1.1.1 Ross--

Table K-1 summarizes the results of the field performance audit conducted at the Ross Incineration Trial Burn, Grafton, Ohio, on June 10 to 14, 1982. The relative percent error (% E) for all audit devices ranged from -3.6 to 2.8%.

TABLE K-1. ROSS PERFORMANCE AUDIT RESULTS

Parameter	Device audited	% E
O_2	Orsat - 2865	+2.8
CO_2	Orsat - 2865	-3.6
O_2	Polarigraph - 7003	0.2
CO_2	Horiba NDIR	1.5
Barometric pressure	Aneroid barometer - 4029	-0.03
Probe temperature	Direct comp.	2.5
Gas volume	Meter box - 9	2.5
Gas volume	Dry gas meter - 2252	-0.1

3.1.1.2 Plant B--

Table K-2 summarizes the results of the field performance audit conducted at Plant B on July 22 and 23, 1982. The relative percent error (% E) for all audit devices ranged from -4.05 to 13.9%. The Orsat O_2 problem (13.9%) was subjected to immediate corrective action procedure by the field QCC; all previous O_2 results were recalculated and the results were determined to be acceptable.

TABLE K-2. PLANT B PERFORMANCE AUDIT RESULTS

Parameter	Device audited	% E
O_2	Orsat, Cyl %/Scott	+13.9
CO_2	Orsat, Cyl %/Scott	+2.8
O_2	Polarigraph 7003	+1.9
CO_2	Horiba NDIR	+2.8
THC	Beckman 402, Hot FID - 8736	Pass[a]
Probe temperature	Direct comp. inlet	+6.2
Stack temp. probe inlet	Direct comp.	+5.8
Gas volume	Meter box - 10	-4.05
Gas volume	Dry gas meter - 8059	+0.8

[a] On scale set to 100 ppm; 1-ppm standard read as 1.8 ppm.

3.1.1.3 Du Pont--

Table K-3 summarizes the results of the field performance audit conducted at Du Pont, La Place, Louisiana, on November 16, 17, and 18, 1982. The relative percent error (% E) for all audit devices ranged from -1.4 to 3.4%.

TABLE K-3. DU PONT PERFORMANCE AUDIT RESULTS

Parameter	Device audited	% E
O_2	Orsat - 2865	+3.4
CO_2	Orsat - 2865	-1.4
O_2	Polarigraph - 7003	0.0
CO_2	Horiba NDIR - 7952	0.0
Barometric pressure	Aneroid barometer - 4029	+0.2
Stack temperature	Meter box - 6	0.0
Sample temperature	Sample box - 5	0.0
Gas volume	Meter box - 6	+1.6
Gas volume	Dry gas meter - 9044	+2.2
Gas volume	Dry gas meter - 8052	+1.6

3.1.2 Systems Audit

Each sampling station at all three plants was audited at least once during the audit period. A total of 38 negative findings were recorded, none of which affected the data quality for the test series.

It was determined that, overall, the sampling operation which was audited was acceptable. The sample handling and resulting sample traceability were also deemed to be acceptable.

3.2 LABORATORY AUDIT

Performance and systems audits for samples obtained from eight plants were conducted: Ross, Upjohn, Plant B, American Cyanamid, Zapata, Mitchell, Du Pont, and TWI.

3.2.1 Performance Audit

The results of the performance audit analysis for volatile POHCs, semivolatile POHCs, metals, and chloride are given in Tables K-4 through K-28. The tables present the individual replicate determinations for each analyte. The average relative percent error, relative percent precision, warning limit range, and control limit range are also given in all tables; these values are reported to two significant figures. The performance audit samples were used as controls; plant samples were not analyzed if the quality control coordinator did not accept the performance audit results.

Relative percent error (% E) for each determination was calculated by the following equation:

$$\% \text{ E} = \frac{\text{RV} - \text{TV}}{\text{TV}} \times 100$$

where RV = reported value
TV = theoretical value

When RV = TV, then the relative percent error is zero, indicating no deviation from theoretical. The average relative percent error was calculated by dividing the sum of the individual relative percent errors by the number of replicates. Relative percent precision was calculated by:

$$\text{Relative percent precision} = \sqrt{\frac{\Sigma X^2 - \frac{(\Sigma X)^2}{n}}{n - 1}}$$

where X = relative percent error
n = number of replicates (for n > 1)

The warning and control limits were based on recommendations by the U.S. EPA. These were calculated by multiplying the relative precision by 2 and 2.56, respectively. These values are statistical in nature and approximate the 95% and 99% confidence intervals.

3.2.1.1 Ross--

The performance audit results for volatile POHCs, semivolatile POHCs, metals, and chlorides are shown in Tables K-4 to K-7, respectively. Summaries of the tables are given below.

- Volatiles
  - Forty-two (42) relative error values were reported for 6 compounds.
  - The average relative error ranged from -9.3 to 8.6%.
  - The relative precision ranged from 4.4 to 11%.
  - One relative error value was outside a warning limit range: carbon tetrachloride (-10%).
  - No relative error value was outside a control limit range.
- Semivolatiles
  - Thirty-one (31) relative error values were reported for 7 compounds.
  - A single relative error value was reported for decafluorobiphenyl (15%).
  - The average relative error ranged from 4.8 to 42%.
  - The relative precision ranged from 2.4 to 49%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.
  - One value for bis(2-ethylhexyl) phthalate (120%) was an obvious outlier but was included in the relative error and precision calculations.
- Metals
  - Sixty-three (63) relative error values were reported for 7 metals.
  - The average relative error ranged from -6.3 to 2.4%.
  - The relative precision ranged from 0.44 to 2.5%.
  - Two relative error values were outside a warning limit range: arsenic (4%) and mercury (3%).
  - No relative error value was outside a control limit range.

- Chlorides

  * Four (4) relative error values were reported.
  * The average relative error was 1.2%.
  * The relative precision was 1.5%.
  * No relative error value was outside a warning limit range.
  * No relative error value was outside a control limit range.

TABLE K-4. ROSS: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

Analyte		Relative error (%)						
	Month:	6	6	7	7	7	7	8
	Day:	25	29	1	2	6	7	22
	Year:	82	82	82	82	82	82	82
Chloroform		-19	0.2	-3	5	7	-10	-2
Carbon tetrachloride		11	8	15	18	5	13	-10
Trichloroethene		1	-11	-8	-5	-14	-12	-8.8
Benzene		1	-9	-11	-5	-10	-10	-21
Tetrachloroethene		9	-2	5	4	3	1	-4
Toluene		1	-7	-9	-6	-13	20	-7.5

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloroform	7	-3.1	9.0	-21 to 15	-26 to 20
Carbon tetrachloride	7	8.6	9.3	-9.9 to 27	-15 to 32
Trichloroethene	7	-8.3	5.0	-18 to 1.8	-21 to 4.6
Benzene	7	-9.3	6.7	-23 to 4.0	-26 to 7.7
Tetrachloroethene	7	2.3	4.4	-6.5 to 11	-8.9 to 14
Toluene	7	-3.1	11	-25 to 19	-31 to 25

TABLE K-5. ROSS: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED SEMIVOLATILE POHCs

Analyte		Relative error (%)				
	Month:	7	7	7	7	8
	Day:	2	6	7	22	11
	Year:	82	82	82	82	82
Phenol		7	10	-17	5	11
Decafluorobiphenyl		15				
D_8-Naphthalene		15	45	-2	11	20
Naphthalene		12	10	-9	9	11
Hexachlorobenzene		9	7	4	6	3
D_{12}-Chrysene		-1	0	20	-8	18
Bis(2-ethylhexyl) phthalate		-5	9	120	46	38

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Phenol	5	4.8	13	-22 to 31	-29 to 39
Decafluorobiphenyl	1				
D-Naphthalene	5	18	17	-17 to 52	-26 to 62
Naphthalene	5	6.6	8.8	-11 to 24	-16 to 29
Hexachlorobenzene	5	5.8	2.4	1.0 to 11	-0.31 to 12
D_{12}-Chrysene	5	5.8	13	-19 to 31	-26 to 38
Bis(2-ethylhexyl) phthalate	5	42	49	-55 to 140	-83 to 170

TABLE K-6. ROSS: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED METALS

		Relative error (%)								
	Month:	7	7	7	7	7	7	7	7	7
	Day:	21	20	21	20	21	20	21	20	20
Analyte	Year:	82	82	82	82	82	82	82	82	82
Arsenic		3	2	2	2	2	2	2	4	3
Barium		-2	-3	-3	-3	-2	-3	-2	-3	-3
Cadmium		0	-2	-1	-1	-2	-1	-2	-3	-1
Chromium		-2	-2	-2	-2	-3	-2	-3	-2	-2
Mercury		-4	-5	-2	-3	-4	-3	-5	3	-4
Lead		-1	-1	-2	-2	-2	-1	-2	-2	-1
Selenium		-8	-5	-5	-7	-5	-7	-6	-7	-7

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Arsenic	9	2.4	0.73	0.99 to 3.9	0.58 to 4.3
Barium	9	-2.7	0.50	-3.7 to -1.7	-3.9 to -1.4
Cadmium	9	-1.4	0.88	-3.2 to 0.32	-3.7 to 0.81
Chromium	9	-2.2	0.44	-3.1 to -1.3	-3.4 to -1.1
Mercury	9	-3.0	2.5	-7.9 to 1.9	-9.3 to 3.3
Lead	9	-1.6	0.53	-2.6 to -0.5	-2.9 to -0.20
Selenium	9	-6.3	1.1	-8.6 to -4.1	-9.2 to -3.5

TABLE K-7. ROSS: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF CHLORIDES

		Relative error (%)			
	Month:	7	7	7	7
	Day:	30	30	30	30
Analyte	Year:	82	82	82	82
Chloride		0	3.4	0.71	0.71

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloride	4	1.2	1.5	-1.8 to 4.2	-2.6 to 5.1

3.2.1.2 Upjohn--

The performance audit results for volatile POHCs, semivolatile POHCs, and chlorides are shown in Tables K-8 to K-10, respectively. Summaries of the tables are given below.

- Volatiles

  - Sixty-two (62) relative error values were reported for 8 compounds.
  - The average relative error ranged from 1.0 to 16%.
  - The relative precision ranged from 9.0 to 42%.
  - Two relative error values were outside a warning limit range: carbon tetrachloride (76%) and toluene (116%).
  - No relative error value was outside a control limit range.
  - One value for toluene (116%) was an obvious outlier but was included in relative error and precision calculations.

- Semivolatiles

  - Fifteen (15) relative error values were reported for 5 compounds.
  - The average relative error ranged from -20 to 18%.
  - The relative precision ranged from 3.1 to 44%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.

- Chlorides

  - Six (6) relative error values were reported.
  - The average relative error was -1.2%.
  - The relative precision was 2.6%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.

TABLE K-8. UPJOHN: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

Analyte		Relative error (%)							
	Month:	8	8	8	8	9	9	9	9
	Day:	27	28	29	31	9	23	24	27
	Year:	82	82	82	82	82	82	82	82
Methylene chloride			-14	12	18	10	7	-6	3
Chloroform		-12	5	16	16	2	-1	-9	18
1,1,1-Trichloroethane			0	12	15	-5	-10	19	12
Carbon tetrachloride		1	20	76	20	13	-15	0	-11
Trichloroethene		1	2	13	27	-14	6	2	14
Benzene		-8	-7	13	17	34	23	15	3
Tetrachloroethene		-7	-2	13	16	-3	2	-2	-9
Toluene		-23	2	12	8	5	2	116	5

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Methylene chloride	7	5.4	12	-19 to 30	-25 to 36
Chloroform	8	4.4	12	-19 to 28	-25 to 34
1,1,1-Trichloroethane	7	6.1	11	-16 to 29	-22 to 35
Carbon tetrachloride	8	13	29	-44 to 70	-60 to 86
Trichloroethene	8	6.4	12	-18 to 30	-24 to 37
Benzene	8	11	15	-18 to 40	-26 to 48
Tetrachloroethene	8	1	9.0	-17 to 19	-22 to 24
Toluene	8	16	42	-68 to 100	-91 to 120

TABLE K-9. UPJOHN: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED SEMIVOLATILE POHCs

Analyte		Relative error (%)		
	Month:	9	9	9
	Day:	8	9	10
	Year:	82	82	82
m-Dichlorobenzene		-4	0	-8
p-Dichlorobenzene		-18	-16	-12
*N*aphthalene		-10	-37	-13
Hexachlorobenzene		18	11	10
Bis(2-ethylhexyl) phthalate		-21	10	65

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
m-Dichlorobenzene	3	-4.0	4.0	-12 to 4	-14 to 6.2
p-Dichlorobenzene	3	-15	3.1	-21 to -9.2	-23 to -7.5
*N*aphthalene	3	-20	15	-50 to 9.6	-58 to 18
Hexachlorobenzene	3	13	4.4	4.3 to 22	1.8 to 24
Bis(2-ethylhexyl) phthalate	3	18	44	-69 to 110	-93 to 130

TABLE K-10. UPJOHN: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF CHLORIDES

Analyte		Relative error (%)					
	Month:	8	8	8	8	8	8
	Day:	30	30	30	30	30	30
	Year:	82	82	82	82	82	82
Chloride		1	-3	-1	1	-6	0

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloride	6	-1.2	2.6	-6.5 to 4.1	-7.9 to 5.6

3.2.1.3 Plant B--

The performance audit results for volatile POHCs, semivolatile POHCs, and chlorides are shown in Tables K-11 to K-13, respectively. Summaries of the tables are given below.

- Volatiles
  - Fifty-eight (58) relative error values were reported for 8 compounds.
  - The average relative error ranged from -17 to 11%.
  - The relative precision ranged from 5.8 to 19%.
  - Two relative error values were outside a warning limit range: chloroform (-24%) and trichloroethene (-11%).
  - No relative error value was outside a control limit range.
- Semivolatiles
  - Sixty-seven (67) relative error values were reported for 11 compounds.
  - A single relative error value is reported for D_{12}-chrysene (36%).
  - The average relative error ranged from -11 to 63%.
  - The relative precision ranged from 3.5 to 100%.
  - Two relative error values were outside a warning limit range: naphthalene (-10%) and bis(2-ethylhexyl) phthalate (330%).
  - No relative error value was outside a control limit range.
  - One value for bis(2-ethylhexyl) phthalate (330%) was an obvious outlier but was included in the calculations for relative error and precision.
- Chlorides
  - Six (6) relative error values were reported.
  - The average relative error was -1.2%.
  - The relative precision was 2.6%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.

TABLE K-11. PLANT B: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

Analyte		Relative error (%)								
	Month:	8	8	8	8	8	8	8	2	2
	Day:	11	12	17	18	21	24	25	1	2
	Year:	82	82	82	82	82	82	82	83	83
Methylene chloride									1	-9
Chloroform		9	9	-13	-24	6.6	-4	1	3	7
Carbon tetrachloride		40	9	34.3	-17	4.3	28	-5	1	2
Trichloroethene		4	5	-2.2	-11	2.5	8	0	6	6
Benzene		19	7	-4.3	-18	2	19	7	-3	30
3,4-Dichloro-1-butene									-5	-28
Tetrachloroethene		-4	2	-1.3	6.5	15	0	2	10	8
Toluene		2	-5	34	4.3	4.9	14	3	4	5

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Methylene chloride	2	-4.0	7.1	-18 to 10	-22 to 14
Chloroform	9	-1.0	11	-23 to 22	-30 to 29
Carbon tetrachloride	9	11	19	-28 to 49	-39 to 60
Trichloroethene	9	2.1	5.8	-9.4 to 14	-13 to 17
Benzene	9	6.5	15	-23 to 36	-31 to 44
3,4-Dichloro-1-butene	2	-17	16	-49 to 16	-58 to 25
Tetrachloroethene	9	4.2	6.1	-7.9 to 16	-11 to 20
Toluene	9	7.4	11	-15 to 30	-21 to 36

TABLE K-12. PLANT B: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED SEMIVOLATILE POHCs

Analyte		Relative error (%)								
	Month:	8	8	8	9	9	9	9	9	9
	Day:	18	19	20	11	15	17	17	17	22
	Year:	82	82	82	82	82	82	82	82	82
m-Dichlorobenzene		1.03	-3	2.7	24	6	6	14	18	25
p-Dichlorobenzene		17	-19	-9.6	-6	-4	-13	-13	5	16
Decafluorobiphenyl		-6.6	-13	-15						7
2,4-Dimethylphenol		-12	-25	-29	23	18	18	33	42	47
1,2,4-Trichlorobenzene									13	20
D-Naphthalene		-25	10	-33						4
Naphthalene		12.7	2.2	10.3	-10	3	3	3	3	12
Diethyl phthalate									1	6
Hexachlorobenzene		-6.5	3.8	-1.9	15	18	18	13	16	23
D_{12}-Chrysene		36.2								
Bis(2-ethylhexyl) phthalate		14.7	330	26.3	65	25	25	9	63	10

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
m-Dichlorobenzene	9	10	10	-10 to 31	-16 to 37
p-Dichlorobenzene	9	-3.0	13	-29 to 23	-36 to 30
Decafluorobiphenyl	4	-6.8	9.8	-26 to 13	-32 to 18
2,4-Dimethylphenol	9	13	28	-43 to 69	-59 to 85
1,2,4-Trichlorobenzene	2	17	5.0	6.6 to 26	3.8 to 29
D-Naphthalene	4	-11	21	-53 to 32	-65 to 43
Naphthalene	9	4.4	6.9	-9.5 to 18	-13 to 22
Diethyl phthalate	2	3.5	3.5	-3.6 to 11	-5.6 to 13
Hexachlorobenzene	9	11	10	-9.2 to 31	-15 to 37
D_{12}-Chrysene	1				
Bis(2-ethylhexyl) phthalate	9	63	100	-140 to 270	-200 to 330

TABLE K-13. PLANT B: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF CHLORIDES

Analyte		Relative error (%)					
	Month:	8	8	8	8	8	8
	Day:	30	30	30	30	30	30
	Year:	82	82	82	82	82	82
Chloride		1	-2	-1	1	-6	0

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloride	6	-1.2	2.6	-6.5 to 4.1	-7.9 to 5.6

3.2.1.4 American Cyanamid--

The performance audit results for volatile POHCs, semivolatile POHCs, metals, and chlorides are shown in Tables K-14 to K-17, respectively. Summaries of the tables are given below.

- Volatiles
  - Fifty (50) relative error values were reported for 10 compounds.
  - A single relative error value was reported for 3,4-dichloro-1-butene (-3%).
  - The average relative error ranged from -3.0 to 10%.
  - The relative precision ranged from 3.5 to 16%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.
- Semivolatiles
  - Thirty-five (35) relative error values were reported for 15 compounds.
  - Single relative error values were reported for 4-aminophenol (3%), decafluorobiphenyl (-1%), D_8-naphthalene (2%), and D_{12}-chrysene (-40%).
  - The average relative error ranged from -54 to 30%.
  - The relative precision ranged from 6.8 to 95%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.
- Metals
  - Thirty-four (34) relative error values were reported for 12 metals.
  - The average relative error ranged from -12 to 7.2%.
  - The relative precision ranged from 0.21 to 14%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.

- Chlorides

  * Eight (8) relative error values were reported.

  * The average relative error was 1.3%.

  * The relative precision was 4.2%.

  * One relative error value was outside a warning limit range: 10.5%.

  * No relative error value was outside a control limit range.

TABLE K-14. AMERICAN CYANAMID: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

Analyte		Relative error (%)					
	Month:	10	11	11	11	11	2
	Day:	28	5	8	24	29	4
	Year:	82	82	82	82	82	83
Methylene chloride		10	-32	4	-3	9	-6
Chloroform		5.7	5	3	-6	6	8
1,1,1-Trichloroethane		14.6	-2	4	-4	26	
Carbon tetrachloride		12	0	20	-17	8	3
Trichloroethene		6	-8	3	-14	1	20
Benzene		28	-16	27	4	6	11
3,4-Dichloro-1-butene							-3
Tetrachloroethene		10	-5	-5	3	2	19
Toluene		6.8	-18	6	6	4	7
Chlorobenzene					2	7	

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Methylene chloride	6	-3.0	16	-34 to 28	-43 to 37
Chloroform	6	3.6	5.0	-6.3 to 14	-9.1 to 16
1,1,1-Trichloroethane	5	7.7	13	-17 to 33	-24 to 40
Carbon tetrachloride	6	4.3	13	-21 to 30	-28 to 37
Trichloroethene	6	1.3	12	-22 to 25	-29 to 32
Benzene	6	10	16	-23 to 43	-32 to 52
3,4-Dichloro-1-butene	1				
Tetrachloroethene	6	4.0	9.3	-15 to 23	-20 to 28
Toluene	6	2.0	9.8	-18 to 22	-23 to 27
Chlorobenzene	2	4.5	3.5	-2.6 to 12	-4.6 to 14

TABLE K-15. AMERICAN CYANAMID: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED SEMIVOLATILE POHCs

Analyte	Month: Day: Year:	Relative error (%) 11 29 82	1 20 83	1 20 83
Aniline		12	14	0
Phenol		135	-51	7
Nitrobenzene		-5	5	8
o-Nitrophenol		-16	-12	3
2-Nitrotoluene		-4	-9	23
Naphthalene		3	-11	-3
2-Aminophenol			-92	-15
Quinoline		8	-1	-20
4-Aminophenol				3
m-Dinitrobenzene		19	-29	8
p-Nitrophenol		-9	13	-16
Bis(2-ethylhexyl) phthalate		-14	-54	
Decafluorobiphenyl			-1	
D_8-Naphthalene			2	
D_{12}-Chrysene			-40	

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Aniline	3	8.7	7.6	-6.5 to 24	-11 to 28
Phenol	3	30	95	-160 to 220	-210 to 270
Nitrobenzene	3	2.7	6.8	-11 to 16	-15 to 20
o-Nitrophenol	3	-8.0	10	-28 to 12	-34 to 17
2-Nitrotoluene	3	3.3	17	-31 to 38	-41 to 47
Naphthalene	3	-4.0	7.0	-18 to 10	-22 to 14
2-Aminophenol	2	-54	54	-160 to 55	-190 to 86
Quinoline	3	-4.0	14	-33 to 24	-41 to 32
4-Aminophenol	1				
m-Dinitrobenzene	3	-1.0	25	-51 to 50	-65 to 64
p-Nitrophenol	3	-4.0	15	-34 to 26	-43 to 35
Bis(2-ethylhexyl) phthalate	2	-34	28	-91 to 23	-110 to 38
Decafluorobiphenyl	1				
D_8-Naphthalene	1				
D_{12}-Chrysene	1				

TABLE K-16. AMERICAN CYANAMID: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED METALS

Analyte		Relative error (%)		
	Month:	11	11	12
	Day:	30	30	3
	Year:	82	82	82
Silver		-1	0	-24
Aluminum		-3	-3	-6.6
Arsenic		-1.3	0	0.5
Barium		-3.7	-3	-3.6
Cadmium		4.6	6.4	2.2
Chromium		-12	-9	-14
Copper		8.9	11	1.6
Iron		-2.4	-2.3	-2.6
Mercury		7.4	3.5	6.1
Lead		4.3	3.8	
Selenium		-2.5	-8.3	-3.1
Zinc		-9.3	6.2	7.1

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Silver	3	-8.0	14	-35 to 19	-43 to 26
Aluminum	3	-4.0	2.1	-8.4 to 0	-9.5 to 1.1
Arsenic	3	0	0.93	-2.1 to 1.6	-2.6 to 2.1
Barium	3	-3.0	0.38	-4.2 to -2.7	-4.4 to -2.5
Cadmium	3	4.4	2.1	0.19 to 8.6	-1 to 9.8
Chromium	3	-12	2.6	-17 to -6.5	-18 to -5.1
Copper	3	7.2	4.9	-2.7 to 17	-5.5 to 20
Iron	3	-2.0	0.21	-2.9 to -2.0	-3 to -1.9
Mercury	3	5.7	2.0	1.7 to 9.6	0.58 to 11
Lead	2	4.1	0.35	3.3 to 4.8	3.1 to 5.0
Selenium	3	-5.0	3.2	-11 to 1.8	-13 to 3.5
Zinc	3	1.3	9.2	-17 to 20	-22 to 25

TABLE K-17. AMERICAN CYANAMID: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF CHLORIDES

		Relative error (%)							
	Month:	11	11	11	11	11	11	11	11
	Day:	19	19	19	19	19	19	19	19
Analyte	Year:	82	82	82	82	82	82	82	82
Chloride		3.3	10.5	0	0.8	-2.1	-2.1	1.8	-2.1

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloride	8	1.3	4.3	-7.2 to 9.7	-9.6 to 12

3.2.1.5 Zapata--

The performance audit results for volatile POHCs are shown in Table K-18. The summary of the table is given below.

- Volatiles
  - Fifty-five (55) relative error values were reported for 9 compounds.
  - A single relative error value was reported for 3,4-dichloro-1 butene (0%).
  - The average relative error ranged from -12 to 13%.
  - The relative precision ranged from 4.9 to 11%.
  - One relative error value was outside a warning limit range: benzene (35%).
  - No relative error value was outside a control limit range.

TABLE K-18. ZAPATA: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

Analyte		Relative error (%)						
	Month:	10	10	10	10	10	10	2
	Day:	4	5	6	8	18	19	7
	Year:	82	82	82	82	82	82	83
Methylene chloride		-16	3	-10	14	1	-17	-9
Chloroform		-18	-16	-17	-5	-5	-16	-4
1,1,1-Trichloroethane		-3	-3	-8	6	17	18	
Carbon tetrachloride		-7	5	-4	2	7	7	-9
Trichloroethene		-9	-1	-5	-2	1	6	-5
Benzene		5	12	10	8	7	35	11
3,4-Dichloro-1-butene								0
Tetrachloroethene		-6	-3	0	-3	10	8	4
Toluene		-15	3	-7	14	10	1	2

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Methylene chloride	7	-5.0	11	-27 to 18	-34 to 24
Chloroform	7	-12	6.5	-25 to 1.4	-28 to 5.1
1,1,1-Trichloroethane	6	4.5	11	-18 to 27	-24 to 33
Carbon tetrachloride	7	0.1	6.7	-13 to 14	-17 to 17
Trichloroethene	7	-2.0	4.9	-12 to 7.6	-15 to 10
Benzene	7	13	10	-7.8 to 33	-13 to 39
3,4-Dichloro-1-butene	1				
Tetrachloroethene	7	1.4	6.1	-11 to 14	-14 to 17
Toluene	7	1.1	9.8	-18 to 21	-24 to 26

3.2.1.6 Mitchell--

The performance audit results for volatile POHCs, semivolatile POHCs, and chlorides are shown in Tables K-19 to K-21, respectively. Summaries of the tables are given below.

- Volatiles
  - One hundred five (105) relative error values were reported for 9 compounds.
  - The average relative error ranged from -14 to 19%.
  - The relative precision ranged from 8.3 to 60%.
  - Three relative error values were outside a warning limit range: methylene chloride (-57%), benzene (206%), and tetrachloroethene (23%).
  - One relative error value was outside a control limit range: benzene (206%); however, it is an obvious outlier.
- Semivolatiles
  - Twenty-two (22) relative error values were reported for 11 compounds.
  - The average relative error ranged from -32 to 1.0%.
  - The relative precision ranged from 1.4 to 40%.
  - No relative error value was outside a warning limit range.
  - No relative error value was outside a control limit range.
- Chlorides
  - Eight (8) relative error values were reported.
  - The average relative error was 1.3%.
  - The relative precision was 4.2%.
  - One relative error value was outside a warning limit range: 10.5%.
  - No relative error value was outside a control limit range.

TABLE K-19. MITCHELL: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

	Relative error (%)											
Month:	11	11	11	11	11	11	11	11	11	11	11	11
Day:	9	10	11	15	16	17	18	18	19	22	30	3
Analyte Year:	82	82	82	82	82	82	82	82	82	82	82	82
Methylene chloride	-30	6	-21	-57	-28	-6	-21	-14	-4	-14	17	7
Chloroform	-2	-4	-10	-22	5	-5	-8	18	20	5	2	13
1,1,1-Trichloroethane	-9	2	-18	-22	7	-8	-1	13	-3	-5	2	14
Carbon tetrachloride	5	6	-14	2	-4	13	28	14	11	13	22	4
Trichloroethene	-6	10	3	-16	0	-5	-3	5	-5	-7	0	15
Benzene	-13	7	-5	-3	-2	-9	8	10	206	18	6	3
Tetrachloroethene	-8	5	-8	5	1	-9	9	14	-2	-7	0	23
Toluene	9	-19	-11	7	6	-13	18	11	-9	3	9	16
Chlorobenzene				8	13	-15	9	9	-7	-3	0	31

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Methylene chloride	12	-14	20	-54 to 26	-65 to 37
Chloroform	12	1.0	12	-23 to 25	-30 to 32
1,1,1-Trichloroethane	12	-2.0	11	-24 to 20	-31 to 26
Carbon tetrachloride	12	8.3	11	-14 to 31	-20 to 37
Trichloroethene	12	-1.0	8.3	-17 to 16	-22 to 20
Benzene	12	19	60	-100 to 140	-130 to 170
Tetrachloroethene	12	1.9	9.9	-18 to 22	-23 to 27
Toluene	12	2.3	12	-22 to 27	-29 to 33
Chlorobenzene	9	5.0	13	-22 to 32	-29 to 39

TABLE K-20. MITCHELL: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED SEMIVOLATILE POHCs

Analyte	Month: Day: Year:	Relative error (%) 11 30 82	 12 1 82
Phenol		-40	3
Isophorone		-26	-7
2,4-Dimethylphenol		3	-1
Naphthalene		0	-9
Dodecanol		-1	-9
Diethyl phthalate		-4	-6
Butylbenzyl phthalate		-2	-50
Bis(2-ethylhexyl) phthalate		-3	-60
Decafluorobiphenyl		4	-17
D_8-Naphthalene		5	-13
D_{12}-Chrysene		4	-51

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Phenol	2	-19	30	-79 to 42	-96 to 59
Isophorone	2	-17	13	-43 to 10	-51 to 18
2,4-Dimethylphenol	2	1.0	2.8	-4.7 to 6.7	-6.2 to 8.2
Naphthalene	2	-5.0	6.4	-17 to 8.3	-21 to 12
Dodecanol	2	-5.0	5.7	-16 to 6.3	-20 to 9.5
Diethyl phthalate	2	-5.0	1.4	-7.8 to -2.2	-8.6 to -1.4
Butylbenzyl phthalate	2	-26	34	-94 to 42	-110 to 61
Bis(2-ethylhexyl) phthalate	2	-32	40	-110 to 49	-140 to 72
Decafluorobiphenyl	2	-7.0	15	-36 to 23	-45 to 32
D_8-Naphthalene	2	-4.0	13	-30 to 22	-37 to 28
D_{12}-Chrysene	2	-24	39	-100 to 54	-120 to 76

TABLE K-21. MITCHELL: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF CHLORIDES

Analyte		Relative error (%)							
	Month:	11	11	11	11	11	11	11	11
	Day:	19	19	19	19	19	19	19	19
	Year:	82	82	82	82	82	82	82	82
Chloride		3.3	10.5	0	0.8	-2.1	-2.1	1.8	-2.1

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloride	8	1.3	4.2	-7.2 to 9.7	-9.6 to 12

3.2.1.7 Du Pont--

The performance audit results for volatile POHCs, semivolatile POHCs, and chlorides are shown in Tables K-22 to K-24, respectively. Summaries of the tables are given below.

- Volatiles
    - ☆ One hundred twenty-five (125) relative error values were reported for 13 compounds.
    - ☆ The average relative error ranged from -14 to 4.2%.
    - ☆ The relative precision ranged from 2.5 to 29%.
    - ☆ One relative error value was outside a warning limit range: methylene chloride (76%).
    - ☆ One relative error value was outside a control limit range: methylene chloride (76%).
- Semivolatiles
    - ☆ One hundred fifteen (115) relative error values were reported for 13 compounds.
    - ☆ The average relative error ranged from -6.3 to 20%.
    - ☆ The relative precision ranged from 8.2 to 63%.
    - ☆ Four relative error values were outside a warning limit range: 3,4-dichloro-1-butene (154%), *trans*-1,4-dichloro-2-butene (29%), phenol (27%), and methylbenzyl chloride (49%).
    - ☆ No relative error value was outside a control limit range.
- Chlorides
    - ☆ Eight (8) relative error values were reported.
    - ☆ The average relative error was 1.3%.
    - ☆ The relative precision was 4.2%.
    - ☆ One relative error value was outside a warning limit range: 10.5%.
    - ☆ No relative error value was outside a control limit range.

TABLE K-22. DU PONT: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

Analyte		Relative error (%)												
	Month:	-	12	12	12	12	12	12	12	12	12	1	1	1
	Day:	-	2	6	8	9	10	13	14	17	15	3	4	5
	Year:	82	82	82	82	82	82	82	82	82	82	83	83	83
Methylene chloride		22	-17	-9	-1	8	-17	3	76	11	-55	-10	-3	-10
Chloroform		0	-3	14	8	3	-11	6	-5	0	-4	18	6	20
1,1,1-Trichloroethane		7	11	14	6	10	-19	-7	10		-4			
Carbon tetrachloride		19	-27	-12	13	8	-31	0	6	3	6	-12	8	-6
Bromodichloromethane										3		1	-3	1
Trichloroethene		0	-1	16	17	8	-8	-4	-1	14	-8	5	3	-2
Benzene		5	0	13	16	-1	-6	19	10	0	10	-13	-4	5
cis-1,4-Dichloro-2-butene										8		-9	-27	-26
Bromoform										-2		4	6	8
3,4-Dichloro-1-butene										0		-2	7	6
Tetrachloroethene		-4	-9	14	12	7	-13	2	-10	8	-4	-4	-5	-3.
Toluene		1	-6	8	5	16	-13	12	1	17	-1	-19	12	-5
Chlorobenzene		4	-13	18	18	13	-15	5	3		-11			

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Methylene chloride	13	0	29	-59 to 59	-75 to 75
Chloroform	13	4.0	9.3	-15 to 23	-20 to 28
1,1,1-Trichloroethane	9	3.1	11	-19 to 25	-25 to 31
Carbon tetrachloride	13	-2.0	15	-32 to 28	-40 to 37
Bromodichloromethane	4	0.5	2.5	-4.5 to 5.5	-5.9 to 6.9
Trichloroethene	13	3.0	8.5	-14 to 20	-19 to 25
Benzene	13	4.2	9.3	-14 to 23	-20 to 28
cis-1,4-Dichloro-2-butene	4	-14	17	-47 to 20	-56 to 29
Bromoform	4	4.0	4.3	-4.6 to 13	-7.1 to 15
3,4-Dichloro-1-butene	4	2.8	4.4	-6.1 to 12	-8.6 to 14
Tetrachloroethene	13	-1.0	8.6	-18 to 17	-23 to 21
Toluene	13	2.2	11	-20 to 24	-26 to 30
Chlorobenzene	9	2.4	13	-23 to 28	-31 to 35

TABLE K-23. DU PONT: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED SEMIVOLATILE POHCs

Analyte	Month: Day: Year:	Relative error (%)								
		1 4 83	1 5 83	1 11 83	1 12 83	1 13 83	1 14 83	1 17 83	1 18 83	1 19 83
3,4-Dichloro-1-butene		19	3	-29		-28	-10	154	28	
trans-1,4-Dichloro-2-butene		8	0	1	29	-10	-12	8	0	3
o-Chlorotoluene		9	12	-5	25	-15	-15	4	-17	-5
Phenol		4	-7	-10	10	-12	-4	27	4	1
Benzyl chloride		11	-9	-2	17	-14	-9	10	-8	6
Hexachloroethane		17	-4	1	13	-13	-11	7	-2	6
Methylbenzyl chloride		6	-9	49	16	-10	-9	-15	1	5
Tetrachlorobutene		14	-2	-1	21	-18	-4	7	1	16
Decafluorobiphenyl		12	2	0	12	-14	-4	12	3	5
D_8-Naphthalene		27	10	3	19	0	-3	20	9	13
Naphthalene		-3	-9	-9	3	-18	-16	7	-9	-3
p-Chlorophenol		9	17	-20	7	33	2	57	28	-1
D_{12}-Chrysene		5	9	26	-2	14	16	-10	13	9

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
3,4-Dichloro-1-butene	7	20	63	-110 to 150	-140 to 180
trans-1,4-Diol-2-butene	9	3.0	12	-21 to 27	-28 to 34
o-Chlorotoluene	9	-0.8	14	-30 to 28	-38 to 36
Phenol	9	1.4	12	-23 to 25	-29 to 32
Benzyl chloride	9	0.22	11	-22 to 22	-28 to 28
Hexachloroethane	9	1.6	10	-19 to 22	-25 to 28
Methylbenzyl chloride	9	3.8	20	-35 to 43	-46 to 54
Tetrachlorobutene	9	3.8	12	-20 to 28	-27 to 35
Decafluorobiphenyl	9	3.1	8.6	-14 to 20	-19 to 25
D_8-Naphthalene	9	11	9.9	-9.0 to 31	-15 to 36
Naphthalene	9	-6.3	8.2	-23 to 10	-27 to 15
p-Chlorophenol	9	15	22	-30 to 59	-43 to 72
D_{12}-Chrysene	9	8.9	11	-12 to 30	-18 to 36

TABLE K-24. DU PONT: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF CHLORIDES

Analyte		Relative error (%)							
	Month:	11	11	11	11	11	11	11	11
	Day:	19	19	19	19	19	19	19	19
	Year:	82	82	82	82	82	82	82	82
Chloride		3.3	10.5	0	0.8	-2.1	-2.1	1.8	-2.1

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloride	8	1.3	4.2	-7.2 to 9.7	-9.6 to 12

3.2.1.8 TWI--

The performance audit results for volatile POHCs, semivolatile POHCs, metals, and chlorides are shown in Tables K-25 to K-28, respectively. Summaries of the tables are given below.

- Volatiles
  - * Two hundred fifty-three (253) relative error values were reported for 14 compounds.
  - * The average relative error ranged from -0.86 to 10%.
  - * The relative precision ranged from 7.8 to 21%.
  - * Eight (8) relative error values were outside a warning limit range: chloroform (-20%), 1,1,1-trichloroethane (-19%), carbon tetrachloride (28%), bromodichloromethane (-25%), trichloroethene (18%), benzene (-28%), bromoform (-46%), and bromochloromethane (-13%).
  - * Four (4) relative error values were outside a control limit range: chloroform (-20%), benzene (-28%), bromoform (-46%), and bromodichloromethane (-25%).
- Semivolatiles
  - * Ninety-five (95) relative error values were reported for 12 compounds.
  - * The average relative error ranged from -14 to 11%.
  - * The relative precision ranged from 7.0 to 18%.
  - * No relative error value was outside a warning limit range.
  - * No relative error value was outside a control limit range.
- Metals
  - * Thirty-eight (38) relative error values were reported for 13 metals.
  - * The average relative error ranged from -15 to 190%.
  - * The relative precision ranged from 0 to 98%.
  - * No relative error value was outside a warning limit range.
  - * No relative error value was outside a control limit range.
  - * Uniformly high selenium values as determined by ICP analysis were most probably caused by spectral interference.

- Chlorides

  * Three (3) relative error values were reported.
  * The average relative error was 5.8%.
  * The relative precision was 0.17%.
  * No relative error value was outside a warning limit range.
  * No relative error value was outside a control limit range.

In summary, an extensive laboratory performance audit program was conducted. A total of 1,308 individual values were obtained, 8 of which were single values. Of the 1,300 values used to determine the warning and control limits, 29 were outside the warning limit ranges and 6 were outside the control limit ranges. Overall, the laboratory analysis phase of the project is judged to be adequate and acceptable.

3.2.2 Systems Audit

The systems audit, which examined the conduct of laboratory practices as stipulated in the Quality Assurance Plan for Laboratory Analysis, indicated that all lab work was acceptable and performed according to protocol. Sample traceability was maintained with no apparent discrepancies.

TABLE K-25. TWI: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED VOLATILE POHCs

Analyte	Relative error (%)																				
Month:	2	2	2	2	2	2	2	2	2	2	2	3	3	3	3	3	3	3	3	3	4
Day:	9	10	11	14	15	16	17	18	22	25	28	2	3	4	7	8	9	10	11	14	26
Year:	83	83	83	83	83	83	83	83	83	83	83	83	83	83	83	83	83	83	83	83	83
Methylene chloride	-5	-18	21	-11	15	-15	21.7	12	9	16	-6	14	12	0	-4	-6	2	-5	10	0	-14
Chloroform	-4	-20	12	8	13	7	7	7	-10	2	3	1	2	7	-2	6	6	8	20	-1	-8
Methylene bromide									4	14	-6	1	9	7	20	-3	9	6	5	2	-8
1,1,1-Trichloroethane									-9	11	-3	-3	7	9	14	5	7	10	5	-9	-19
Carbon tetrachloride	-13	-28	0	-4	-12	5	-12	22	1	-4	4	4	9	4	25	0	18	10	14	9	-23
Bromodichloromethane		-16	6	5	14	-5	0	4	-7	8	0	4	-6	9	9	-3	9	3	2	3	-25
Trichloroethene	-5	-14	0	-8	8	-14	-3	-2	-2	1	-6	-3	-8	5	18	6	0	9	11	5	-16
Benzene	-10	-28	12	11	14	0	2	-6	-2	9	-5	6	4	11	17	-4	15	2	19	10	4
Bromoform	7	-7	37	7	48	14	22.5	31	-46	28	38	-8	-3	-3	16	-7	12	8	16	7	-5
Tetrachloroethene	-2	-17	8	-6	10	-3	20.5	3	-8	4	-4	8	1	5	13	9	23	-2	11	0	-13
Toluene	-7	-15	13	1	3	-9	1	-4	-12	3	-13	10	-2	15	17	-5	-1	-5	9	6	-15
Chlorobenzene									-5	3	0	-14	-1	10	14	-7	11	3	11	5	-12
Bromochloromethane									-8	13	5	-4	6	14	10	-1	8	7	7	10	-13
Dibromochloromethane									5	11	19	-2	-2	22	3	12	9	12	3	-6	-15

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Methylene chloride	21	2.3	12	-22 to 27	-29 to 33
Chloroform	21	3.0	8.7	-14 to 21	-19 to 25
Methylene bromide	13	4.6	7.8	-11 to 20	-15 to 25
1,1,1-Trichloroethane	13	1.9	9.7	-18 to 21	-23 to 27
Carbon tetrachloride	21	1.4	14	-26 to 29	-34 to 36
Bromodichloromethane	20	0.70	9.2	-18 to 19	-23 to 24
Trichloroethene	21	-0.86	8.7	-18 to 17	-23 to 22
Benzene	21	3.9	11	-18 to 26	-24 to 32
Bromoform	21	10	21	-31 to 52	-43 to 63
Tetrachloroethene	21	2.9	10	-17 to 23	-23 to 29
Toluene	21	-0.48	9.7	-20 to 19	-25 to 24
Chlorobenzene	13	1.4	9.0	-17 to 19	-22 to 24
Bromochloromethane	13	4.2	8.2	-12 to 21	-17 to 25
Dibromochloromethane	13	5.5	10	-15 to 26	-21 to 32

TABLE K-26. TWI: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED SEMIVOLATILE POHCs

Analyte		Relative error (%)							
	Month:	2	2	2	2	2	2	2	3
	Day:	16	17	18	22	23	25	28	1
	Year:	83	83	83	83	83	83	83	83
Decafluorobiphenyl		-16	3	-12	-14	-9	-18	6	-3
1,2,4-Trichlorobenzene		-21	3	-14	-10	-8	-16	9	-1
Naphthalene		-5	13	-2	2	6	-5	18	-12
D_8-Naphthalene		-18	-13	-25	-9	-21	-11	-6	4
Hexachlorobutadiene		-12	7	-3	-9	-1	-14	14	1
Hexachlorocyclopentadiene		-1	24	1	13	1	20	19	10
Octachlorocyclopentene		-2	21	-5	8	-1	0	14	12
Hexachlorobenzene		-8	1	-8	-2	-5	-12	9	4
Dibutyl phthalate		12	8	-8	-3	-8	-4	-19	-15
Chlordane			9	2	27	-2	8	-8	-1
D_{12}-Chrysene		20	-1	-7	0	-9	-8	-18	-28
Bis(2-ethylhexyl) phthalate		18	-2	-14	-13	-14	-13	-30	-43

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Decafluorobiphenyl	8	-8.0	8.9	-26 to 10	-31 to 15
1,2,4-Trichlorobenzene	8	-7.0	10	-28 to 13	-33 to 19
Naphthalene	8	1.9	10	-18 to 22	-24 to 28
D_8-Naphthalene	8	-12	9.1	-31 to 5.9	-36 to 11
Hexachlorobutadiene	8	-2.0	9.6	-21 to 17	-27 to 22
Hexachlorocyclopentadiene	8	11	9.7	-8.6 to 30	-14 to 36
Octachlorocyclopentene	8	5.9	9.3	-13 to 24	-18 to 30
Hexachlorobenzene	8	-3.0	7.0	-17 to 11	-21 to 15
Dibutyl phthalate	8	-5.0	11	-26 to 16	-32 to 22
Chlordane	8	5.0	11	-18 to 28	-24 to 34
D_{12}-Chrysene	8	-6.0	14	-34 to 22	-42 to 30
Bis(2-ethylhexyl) phthalate	8	-14	18	-50 to 22	-60 to 32

TABLE K-27. TWI: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF SELECTED METALS

Analyte	Month: Day: Year:	Relative error (%) 3 23 83	 3 23 83	 3 23 83
Calcium		1	2	
Aluminum		-3	-2	-1
Arsenic		11	5	10
Barium		0	3	2
Cadmium		13	50	19
Chromium		16	17	17
Copper		1	15	12
Iron		1	1	1
Mercury		7	11	-62
Lead		-11	-11	16
Selenium		300	130	131
Zinc		8	22	10
Tin		71	17	15

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Calcium	2	1.5	0.7	0.09 to 2.9	-0.3 to 3.3
Aluminum	3	-2.0	1.0	-4.0 to 0	-4.6 to 0.56
Arsenic	3	8.7	3.2	2.2 to 15	0.44 to 17
Barium	3	1.7	1.5	-1.4 to 4.7	-2.2 to 5.6
Cadmium	3	27	20	-12 to 67	-24 to 78
Chromium	3	17	0.58	15 to 18	15 to 18
Copper	3	9.3	7.7	-5.4 to 24	-9.5 to 28
Iron	3	1.0	0	1.0 to 1	1.0 to 1.0
Mercury	3	-15	41	-97 to 67	-120 to 90
Lead	3	-2.0	16	-33 to 29	-42 to 38
Selenium	3	190	98	-8.7 to 380	-64 to 440
Zinc	3	13	7.6	-1.8 to 29	-6.1 to 33
Tin	3	34	32	-29 to 98	-47 to 120

TABLE K-28. TWI: RESULTS OF THE PERFORMANCE AUDIT ANALYSIS OF CHLORIDES

Analyte		Relative error (%)		
	Month:	3	3	3
	Day:	8	8	8
	Year:	83	83	83
Chloride		6	5.7	5.7

Analyte	n	Average relative error (%)	Relative precision (%)	Warning limit range (%)	Control limit range (%)
Chloride	3	5.8	0.17	5.5 to 6.2	5.4 to 6.2

SECTION 4.0

CONCLUSIONS

Overall, these audits indicate that the performance and conduct of the field and laboratory work can be judged to be adequate and acceptable.

More specifically:

- The field performance audit determined that the equipment was calibrated and that it performed adequately.
- The field systems audit determined that the protocols and procedures were adequately followed.
- The volatile and semivolatile POHC analysis was accurate and precise.
- The trace metals analysis was accurate and precise with the exception of selenium analysis for TWI.
- The chloride analysis was accurate and precise.
- Laboratory procedures were performed in an acceptable manner.

APPENDIX L

INTERNAL QUALITY ASSURANCE PROGRAM

SECTION 1.0

INTRODUCTION

This appendix presents the results of the internal QA activities associated with analyses of samples from the eight incinerator test burns for POHCs and for metals. The purpose of generating this internal QA data was to help define the quality of the analytical data. The internal QA data reported here are grouped by incinerator site. The QA data from each site are presented according to type of analytes: volatile POHCs, semivolatile POHCs, and metals. Generally, for each type of analyte there are precision data (resulting from analyses of replicate samples) and accuracy data (obtained from analyses of samples fortified with analytes and/or surrogate compounds).

Other QA activities are discussed in the analytical methods appendix and cover such topics as instrument calibration and performance criteria, preparation and measurement of calibration standards, criteria for mass spectral ion abundance, etc.

In addition, external quality assurance data from the analysis of blind check samples which were submitted for analysis by the Quality Assurance manager are presented in Appendix K. The purpose of daily analysis of blind check samples was to ensure that the GC/MS system (for organics) and ICAP instrument (for metals) were generating data which provided accurate identification and quantification of the analytes. The results of these blind check sample analyses are extremely important data with respect to the analysis of samples and correctly interpreting the analytical results. However, these external check samples do not reflect sample matrix effects which do appear in some of the internal QA data presented in the following sections.

The QA data involving spiked replicates of single samples were generated using samples from Run 2 or Run 3 conducted at each of the incinerator sites. However, the QA data are expected to be representative for all the runs from each site.

Prior to the start of the program, desired ranges were established for the precision and the accuracy of the quality assurance data. However, it was anticipated that these precision and accuracy goals may be overly stringent since nonvalidated methods were being applied, in many cases, to extremely complex sample matrices. The desired ranges for the quality assurance data were the following:

Precision: % RSD ≦ 30%
Accuracy: 70% ≧ Recovery ≦ 130%

A large body of internal QA data was generated during the program. At least some precision data were compiled for 16 different volatile organic compounds, 43 different semivolatile organic compounds, and 12 different metals. At least some accuracy data were compiled for 12 different volatile organic compounds, 37 different semivolatile organic compounds, and 20 different metals.

The internal QA results for the volatile organic compounds were found to be generally better than for the semivolatile organic compounds.

A discussion of how the precision and accuracy data are calculated is presented below. This discussion is followed by a general discussion of the internal QA, followed by a discussion of the internal QA data from each individual plant.

1.1 PRECISION DATA

The precision data for both organics and metals were obtained by analyzing actual trial burn samples in replicate (generally triplicate). The precision is measured by the percent relative standard deviation (% RSD) which is calculated according to

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Precision can be adversely influenced by variations in sample preparation activities, variations in the measurement activities (GC/MS/computer parameters), and unanticipated sample matrix effects.

1.2 ACCURACY (RECOVERY) DATA

Two types of recovery experiments produced the data given in the following sections of the appendix which deal with accuracy. First during analysis for volatile and semivolatile POHCs, all analytical and QA samples were fortified with known amounts of surrogate compounds. The surrogates used, D_4-1,2-dichloroethane and D_{10}-ethylbenzene for volatiles analysis and decafluorobiphenyl, D_8-naphthalene, and D_{12}-chrysene for semivolatiles analysis, do not occur as native sample components because, except for the decafluorobiphenyl, they are isotopically labeled. Fluorinated compounds are generally chemically inert, thermally stable, volatile, easy to chromatograph, and not found in nature except in effluents from fluorocarbon plants. The accuracy for the surrogate compounds is measured by the percent recovery calculated by

$$\text{Percent Recovery} = \frac{\text{Amount Found}}{\text{Amount Fortified}} \times 100$$

Generally, the volatile and semivolatile surrogate spike levels in the waste samples were 250 µg/g. The aqueous spiking levels were 20 µg/L and 20 µg of the semivolatile surrogates were spiked onto XAD. Since the surrogates were spiked into the samples prior to sample preparation and before any screening of the samples prior to GC/MS analysis, it sometimes happened that the concentration of the surrogates in the aliquot of sample analyzed diverged from the optimum analytical concentration range. During analyses of complex samples, which needed to be further diluted, only a small amount of an organic surrogate compound (e.g., ≦ 2 ng) may be presented to the detector during GC/MS analysis. In such situations (measuring amounts of a compound near the detection limit) the error in measuring the surrogate increases, and consequently errors in determining the percent recovery are likely to be greater than when analyzing samples with surrogate concentrations well above their detection limits. Thus, the recoveries of native compounds present at high levels may be quite good, even though the measurement of the surrogates recoveries may not be good.

Second, after analysis of portions of waste feed samples for POHCs and metals, additional aliquots of the samples were spiked in triplicate with selected volatile and semivolatile POHCs; the samples were then prepared as usual and analyzed. Spiking levels for selected analytes were usually chosen to be 10 times the estimated detection limit for compounds not detected during the initial analyses or 2 times the native amount found in the sample. In some cases where the analyte(s) was extremely concentrated, one times the native amount found was spiked into the sample for the fortification experiment.

Recovery of organic analytes from spiked waste samples was calculated according to the following:

$$\%\ \text{Recovery} = \frac{\text{Amount Found in Fortified Sample - Native Amount}}{\text{Amount Fortified}} \times 100$$

Further, in order to monitor recovery of semivolatile analytes from water and XAD, portions of double-distilled water and portions of fresh clean XAD resin were spiked with selected analytes. The water or XAD portions were then extracted and analyzed in the same manner as samples. Recoveries were calculated in the same way as for added surrogate compounds:

$$\%\ \text{Recovery} = \frac{\text{Amount Found}}{\text{Amount Fortified}} \times 100$$

Recovery of metals from fortified samples was calculated the same way as fortified organic analytes. However, because of the large number of metals determined in the samples, the metals were fortified into samples and prepared at the same time that the unfortified samples were prepared. Thus, the fortification level for metal analytes was not based on the native amount found in the sample, and in some cases, the metal fortification levels did not fall into the desired range of 1 to 2 times the native sample level discussed above.

Note that for stack effluent volatile samples the procedure was slightly different and no "internal QA data" are presented for these samples in this appendix. The procedure used for analysis of the volatiles from the stack effluent involves obtaining response factors for the individual analytes by spiking a blank Tenax/charcoal trap with known quantities of internal calibration standards and the analytes of interest. The same internal standards are then spiked onto the actual sample trap just prior to analysis and the sample is quantified based on the response factor established from the blank. Consequently, precision and accuracy data are not presented in this appendix for volatiles analysis of the stack effluent.

SECTION 2.0

GENERAL INTERNAL QUALITY ASSURANCE DATA AND DISCUSSION

This section of the appendix presents summaries of the QA data and includes a general discussion of results obtained. Note that all the QA data are presented, tabulated, and discussed. At this time, no attempt has been made to remove outliers from the data set which may have resulted from such problems as analyte concentrations near the detection level or outside the linear range of the analytical instrument. Furthermore, data were obtained for a large range of analytes; the reported data are not restricted to the specific POHCs of interest for individual incinerator test sites. This fact must be kept in mind when reviewing a general summary of the body of data. In other words, although it is useful to overview the body of QA data as a means of identifying general trends and problem areas, the QA data relevant to each test location should be viewed individually.

Because of the extremely large amount of internal QA data, it is not possible to completely generalize the results. However, several summary tables of the data have been compiled to help evaluate the data; these summary tables are presented in the following sections.

2.1 VOLATILE ORGANIC COMPOUNDS

2.1.1 Precision Data

A summary of the precision data is presented in Table L-1; this summary shows the percentage of the precision data that were within the desired range of ≦ 30% RSD for the volatile organic compounds. The surrogate compounds were spiked into all the samples prior to analysis, while selected samples, both unspiked and spiked, were analyzed in triplicate to generate the precision data for the other compounds. (Each precision measurement represents a set of triplicate values.) The data in Table L-1 show that the repeatability of the analyses for the volatile compounds is very good with most of the measurements falling within the desired range. The complex organic liquid and drum waste feed samples did not cause poorer analytical reproducibility compared to the aqueous wastes.

2.1.2 Accuracy Data

The accuracy, as determined by the percent recovery of spiked analytes within the desired recovery range of 70 to 130% is presented in Table L-2 for the surrogate compounds and for the volatile POHCs used in spiking experiments.

Of the 118 total accuracy measurements made on the aqueous waste, 57 (48%) were within the desired range. Fourteen (12%) of the measurements yielded greater than 130% recovery. Sixteen (14%) of the measurements were within the 50-70% range, and 31 (26%) were within the 0-50% recovery range.

A total of 303 accuracy measurements were made on volatile compounds spiked into the organic waste. Two hundred (66%) of the recovery measurements were within the desired range. Forty-three (14%) of the recovery values were high (> 130% recovery). Thirty-seven (12%) of the recovery values were within the 50-70% range and only 23 (~ 8%) of the measurements were in the 0-50% recovery range.

Of the high values (> 130% recovery), 47% of the aqueous waste high values were due to the surrogate compound D_4-1,2-dichloroethane. This same surrogate accounted for 29% of the high values determined in the organic waste. These results suggest that a different early-eluting volatile surrogate should be used because the mass spectral fragment ion used for this compound is subject to interferences by other compounds.

The accuracy measurements may be affected by several factors including genuine poor recovery of the analyte from some sample matrices, mass spectral interference from coeluting compounds, weak mass spectral signals in highly diluted samples, etc. Inaccuracies in the measurement of the recovery may occur when one attempts to measure quantities of analyte at concentrations outside the upper and lower linear response range of the GC/MS. These limits are compound-specific, are very diverse, and can only be roughly predicted from the response factors and detection limits of a given compound during analysis of calibration standards. When a compound is already present in high concentration, addition of a "spike" may push the concentration beyond the range of linear measurement. Very low native concentrations, on the other hand, are susceptible to errors in measurement which may result in erroneously high recovery data when the spiked samples are analyzed. Another problem, which may be more serious with respect to the analysis of wastes because it is less predictable, occurs when chemical reactions occur within the waste mixtures which may generate or destroy some of the analyte being analyzed. The recovery data for analytes from some samples suggest that this phenomenon may be occurring. In some cases when a sample was reanalyzed after spiking the results obtained were either much lower or higher than for the unspiked sample; reactions within the sample matrix to either decompose or generate the analyte is a plausible explanation. For example, for the Du Pont site, spiked samples of cis-1-4-dichloro-2-butene gave recovery values of 0%; this compound hydrolyzes in the presence of water and it is suspected that this reaction is the reason for the low recoveries. This question merits further study and perhaps modification of some of the sample handling and analysis protocols. For example, it may be necessary to (1) store reactive wastes in the cold and analyze them as soon as possible, and (2) to reanalyze an unspiked waste the same day as the spiked samples in order to observe any changes which may have occurred.

Another aspect to consider is that the samples from certain plants are responsible for a higher proportion of the values determined to be out of the desired range than from other plants. For example, for the very complex Upjohn liquid waste samples, the recoveries of the spiked POHCs were high outside the desired range in all samples, while the recovery of the D_{10}-ethylbenzene surrogate was low in all samples.

The overall recovery data for the organic liquid and drum waste samples was somewhat better than for the aqueous waste. Some of the spiking experiments performed on the aqueous wastes were apparently performed at concentrations which exceeded the water solubility of the spiked compounds resulting in low recoveries. The aqueous wastes contained only very low native organic concentrations which were not high enough to enhance solubilization of the spiked compounds. The spiking of relatively high levels of organics in the aqueous waste from the TWI test site is an example of a situation where this phenomenon is suspected (see Table L-82).

2.2 SEMIVOLATILE ORGANIC COMPOUNDS

2.2.1 Precision Data

A summary of the precision data, showing the percentage of the data that were within the desired relative standard deviation range of < 30% for semivolatile organic compounds is presented in Table L-3. The surrogate compounds were spiked into all the samples prior to extraction, while selected samples, both unspiked and spiked, were analyzed in triplicate to generate the precision data for the other compounds. Only very limited data are available for the surrogate ^{13}C-phenol since it was not used until near the end of the program.

Most of the precision measurements were within the desired range. For example, 65% of all the precision measurements for the aqueous waste were within the desired range, 79% of all the precision measurements for the organic waste were within the desired range, 91% of all the precision measurements for the aqueous samples were within the desired range, and 66% of all the precision measurements for the XAD samples were within the desired range. Since the precision of triplicate sequential analysis of the same sample extract by GC/MS frequently is in the 10-30% RSD range and even higher for some compounds (see Tables L-4 and L-5), the overall precision associated with spiking, preparing, and analyzing triplicate samples is quite good.

For example, Table L-4 presents the results of repeated sequential analysis for semivolatile POHCs of a single sample extract from the Ross site. These data were obtained in order to have some measure of the run-to-run variation in results of GC/MS analyses. Eight of the 10 compounds measured have a precision within the desired acceptance range but the precision for two other compounds, D_{12}-chrysene and bis(2-ethylhexyl) phthalate is very poor (RSD = 69 and 72%, respectively). Inspection of the raw data for these analyses indicate that the mass spectrometer tuning may have been unstable causing quantification biases.

Table L-5 presents the results of repeated sequential GC/MS analysis of a single extract of organic waste No. 301 from the Plant B site along with the results of the original analysis of the same extract. The precision for three sequential analyses is good to excellent with all RSDs within the desired range and most RSDs less than 10%. When the results of the original analyses are included in the calculated averages and relative standard deviations, most of the latter are still within the desired range (RSD ≤ 30%)

and only two compounds, butylbenzylphthalate and the surrogate D_{12}-chrysene show undesirably high RSDs of 45 and 43%, respectively. The butylbenzylphthalate imprecision may be due to contamination with this common environmental contaminant. The imprecision for D_{12}-chrysene may be due to some slight difference in the tuning of the mass spectrometer or possibly due to actual decrease in the sample content of D_{12}-chrysene due to chemical reaction or physical adsorption onto solid surfaces. The results presented in this table give some measure of normal run-to-run and day-to-day variation in the results of quantitative analysis of semivolatile analytes with the GC/MS system.

2.2.2 Accuracy Data

Table L-6 presents the accuracy data (percent recovery of spiked compounds) for the semivolatile organic compounds. The accuracy of the semivolatile analyses is generally not as good as for the volatile analyses. The preparation of samples for analysis of semivolatile organic compounds requires more steps, i.e., extraction of the sample with a solvent followed by evaporative concentration of the solvent extract (extract concentration was not always required for highly concentrated samples such as liquid organic wastes). For the aqueous waste, 58% of all the individual values were in the desired range. Another 10% of the values were in the 50-70% recovery range, while 12% of the recovery values were greater than 130% and 20% of the values were in the 0-50% recovery range. For the organic waste, 34% of all the individual values were within the desired range. Another 28% of the values were high (> 130% recovery). A total of 27% of the decafluorobiphenyl surrogate values were high outside the desired range and 39% of the D_8-naphthalene values were high outside the desired range. None of the D_{12}-chrysene values were high outside the range; however, 57% of the D_{12}-chrysene values showed 0-50% recovery. It appears that in complex organic wastes, D_{12}-chrysene shows a propensity for loss from the sample and the two other surrogates show some propensity for interference in complex waste samples.

It would be desirable to use additional or perhaps different surrogate compounds in future incineration tests. Ideally, the surrogates would be stable isotopically labeled forms of each of the POHCs selected for DRE determination at a trial burn site. However, this is probably not practical; nonetheless, it would be useful to expand the list of surrogate compounds to include labeled forms of commonly observed POHCs such as chlorinated benzenes, phthalates, chlorinated pesticide, etc.

The recoveries of the spiked compounds from water were quite good. A total of 55% of all the individual values were within the desired range. Another 20% of the values were within the 50-70% recovery range. Only 4% of the values were high outside the range.

The recoveries of spiked compounds from XAD were also quite good. A total of 46% of all the individual recovery measurements were within the desired range, while another 22% were within the 50-70% recovery range. Fourteen percent of the values were high out of the desirable range. Most of these high values were accounted for by phenol (56% of total values) and naphthalene (45% of the total values). These two compounds can apparently be solvent-extracted to some extent even from freshly prepared XAD.

There is considerable evidence that the desired recovery ranges (70-130%) selected for this program were much too stringent for difficult sample matrices such as hazardous waste samples. Also, different compounds can be expected to give different recoveries. The document entitled "Uniform Set of Procedures for Trace Organics Analysis, Documentation of Methods, and Verification of Sample Data Generated" has been used by EPA Region VII to establish program QA guidelines for water sample analysis. This document provides method check analysis acceptance criteria for the percent recovery limits of semivolatile organic compounds from water. The recovery acceptance criteria for some of the compounds are listed in Table 7; as can be seen from this table, the range of recoveries is quite wide and varies from an average of 38 to 210%. Although the QA acceptance criteria for Region VII's program is not necessarily directly applicable to this program, it does give an indication of the range of recoveries which might be normally expected. Note that a total of about 85% of all the percent recovery values for semivolatile POHCs and surrogates from all the matrices tested in this study, including aqueous waste, organic/drum waste, water, and XAD, fall within the recovery range of 38 to 210%.

2.3 METALS

2.3.1 Precision Data

The precision data for the metals cannot be compiled into succinct tables as easily as for the organics. Frequently, samples were only analyzed in duplicate, which makes calculation of percent relative standard deviation values less meaningful. The values that were calculated, however, frequently had percent relative standard deviations in the 10-40% range, similar to that observed for the organic analyses. The precision data for replicate measurements of metals in samples from the different incineration tests are given in Tables L-19, L-20, L-36, L-37, L-60, L-77, L-89, and L-90.

2.3.2 Accuracy Data

Because of the larger number of analytes and the relatively less importance of the data with respect to the overall program, duplicate samples were spiked blindly and prepared and analyzed at the same time as the unknown samples. This procedure resulted in many of the metals not being spiked at the 1-2 times native level. However, in situations where the metals were spiked at appropriate levels, the recoveries were generally quite good (60-100% range) from the waste samples. The accuracy data for measurement of recovery of metals spiked into samples from the different incineration tests are given in Tables L-21, L-22, L-23, L-24, L-38, L-39, L-40, L-61, L-78, L-91, and L-92.

SECTION 3.0

QA DATA FOR SPECIFIC INCINERATION SITES

3.1 QA DATA FROM ROSS SITE SAMPLES

3.1.1 Volatile Organic Analytes

3.1.1.1 Precision Data--

Table L-8 gives the results of analyses of replicate extracts of aqueous waste No. 303 from the Ross site for volatile POHCs. The precision of analysis for the three POHCs found (toluene, methylene chloride, and methyl ethyl ketone) is good (RSDs = 0 to 21%).

Table L-9 presents the results of analysis of replicate extracts of organic waste No. 304 from the Ross site for volatile POHCs. Six POHCs were determined and precision for five of the determinations was excellent (RSD $\leqq$ 10%). Somewhat greater variation in results was found for 1,1,1-trichloroethane but the precision is still within the desired acceptance range.

3.1.1.2 Accuracy Data--

Table L-10 gives the recoveries of selected volatile POHCs spiked into replicate samples of aqueous waste No. 303 from the Ross site. Benzene and D_{10}-ethyl benzene showed high recoveries, but acceptable precision. The surrogate compound D_4-1, 2-dichloroethane gave too high recovery and also the precision was outside the desired acceptance range. Toluene gave good recovery but with poor precision. The other analytes gave excellent recoveries (98 - 110%) and precision (RSDs = 2 to 25%).

Table L-11 presents the recoveries of selected volatile POHCs spiked into replicate samples of organic waste No. 304 from the Ross site. Overall, the recoveries are good and the precision is excellent. Benzene and the two deuterated surrogates gave recoveries outside the desired acceptance limits. Their quantification may have been distorted by coeluting organic compounds from the organic waste. The recovery of toluene appears to be low. This effect is probably caused by measuring a very large amount of analyte in the sample and comparing it to a small amount of analyte in the calibration standard (nonlinear response effect).

Table L-12 presents a summary of all the recoveries of the volatile surrogate compounds D_4-1,2-dichloroethane and D_{10}-ethylbenzene spiked into samples from the Ross site. Overall, the recoveries are excellent ranging from 74 to 110%. One recovery for D_{10}-ethylbenzene is high (140%) and outside the desired acceptance range. The surrogate D_4-1,2-dichloroethane was not detected in some samples because it was necessary to take very small aliquots of some sample extracts (organic waste and drum waste composites) for analysis and this resulted in dilution of the D_4-1,2-dichloroethane to the extent that it was not detectable.

3.1.2 Semivolatile Organic Analytes

Table L-13 gives the results of analysis for semivolatile analytes in replicate extracts of organic waste No. 304 from the Ross site. The results were somewhat variable, but overall the precision was good. The precision for bis(2-ethylhexyl)phthalate was outside the desired acceptance range. Interference by coeluting compounds made it impossible to measure phenol and 2,4-dimethyl phenol in all the samples leading to incomplete precision data.

Table L-14 gives the results of analysis for semivolatile analytes in replicate extracts of aqueous waste No. 303 from the Ross site. The precision is poor for several of the analytes. The variation in results may have been caused by differences in procedures used to analyze the samples which make these not truly replicate analyses. The first is that sample 303-1 was taken from a different sample container than samples 301-2 and 301-3. The other problem is that sample 301-1 was analyzed on a different GC/MS instrument than samples 301-2 and 301-3 during the process of changing the program activities from one instrument to another. Neither of these two procedural problems occurred in subsequent tests.

Table L-15 presents the recovery data from analyses for semivolatiles in replicate spiked samples of sample No. 303 from the Ross site. The accuracy and precision for phenol, naphthalene, dimethylphthalate, 1,2,4-trichlorobenzene and the surrogate decafluorobiphenyl are within acceptable ranges. N,N-Dimethylacetamide and the surrogate D_8-naphthalene gave recoveries that are (high) outside the desired acceptance range but with good reproducibility. Poor reproducibility and too-high recovery were found for 2,4-dimethyl phenol and poor reproducibility with too-low recovery were found for the surrogate D_{12}-chrysene. The reason for the departures from the desirable acceptance range for these compounds in this set of spiked samples is not known.

Table L-16 presents the recovery data for semivolatile compound from analyses of replicate spiked organic waste No. 304 from the Ross site. The precision of the determinations was good for all compounds except the surrogate D_{12}-chrysene. However, recoveries for all the analyte compounds are (high) outside the desired acceptance range. The reason for the too-high recovery values is not known, but concentrated mixtures of organic compounds generally give rise to numerous non-analyte chromatographic peaks which may interfere with accurate measurement of the selected ions of the analyte compounds.

Table L-17 gives the recoveries of selected semivolatile analytes from duplicate spiked water and duplicate spiked XAD. The recoveries from water for most of the analytes is very good. Phenol and N,N-dimethyl acetamide show poor recoveries from water, 42% and 3.8% respectively. Both compounds are very soluble in water and are not efficiently extracted by methylene chloride. Bis-(2-ethylhexyl)phthalate also shows a low recovery for unknown reasons. Almost all the recovery determinations from water show precision that is within the desired acceptance range. The results for phenol and N,N-dimethylacetamide are outside the desired acceptance range.

Overall the recoveries of the selected analytes from spiked XAD are very good with the exception of N,N-dimethylacetamide (65%) and D_{12}-chrysene (40%). The precision of the recoveries is overall rather poor with results for most compounds being outside the desired acceptance range. The reason for the variable results is not known.

Table L-18 gives the recoveries of semivolatile surrogate compounds from all samples. A number of the recoveries for decafluorobiphenyl are low. Low recoveries may result for samples whose extracts required extensive concentration and is attributed to volatility losses during sample workup. High recoveries of decafluorobiphenyl were found for two samples of "waste-to-chamber" samples. These high recoveries are probably due to interference from other compounds in the waste mixture.

D_8-naphthalene was found to have somewhat low recoveries for many of the samples from this site. The recoveries for D_{12}-chrysene are mostly within the desired acceptance range with a few samples, notably the spiked waste samples, having low recoveries. The reason for this is not known.

3.1.3 Metals

Table L-19 presents the results of replicate analyses for metals in aqueous waste No. 303 from the Ross site. The reproducibility of most of the determinations is very good. It was not possible to calculate the analysis precision for chromium, lead, or tin because the concentrations of these elements were very near their detection limits.

Table L-20 gives the results of replicate analyses for metals in organic waste No. 304 from the Ross site. The reproducibility of the determinations for most of the analytes is within the desired acceptance limits except for antimony and thallium. The reason for the imprecision for these two elements is not known.

Table L-21 gives the recovery data for metals spiked in triplicate into organic waste No. 304 from the Ross site. Twelve elements were spiked into the waste. Six showed good recoveries in the desired acceptance range. Of the other elements, silver was probably lost through precipitation of silver chloride. Barium, antimony, chromium and lead were spiked at levels which were too low to produce valid recovery data. Selenium shows recoveries which are high and outside the desired acceptance.

Table L-22 gives the recovery data for metals spiked into aqueous waste No. 303 from the Ross site. The analytes silver and arsenic were not detected in the spiked samples. The other spiked elements all showed recoveries which are high and outside the desired acceptance range.

Table L-23 gives the recovery data for metals spiked into scrubber effluent No. 310 from the Ross site. Because of the high concentrations of some elements, e.g., lead, the samples had to be diluted before analysis and this led to rather high detection limits. In the cases of thallium and arsenic, the detection limit was above the spiking level. Silver probably precipitated out of the sample as silver chloride during the sample preparation. Recoveries for the other elements are within the desired acceptance range.

Table L-24 presents the recovery data for metals spiked into drum waste composite No. 527 from the Ross site. Silver probably was lost due to precipitation of silver chloride during the sample preparation. Arsenic, mercury, antimony, selenium, and thallium were spiked at levels very near the detection limit, and thus any recovery data generated would not be valid. Very low recoveries were found for beryllium, cadmium, and lead, while the recovery of nickel is high and outside the desired acceptance range. Barium and chromium gave recoveries within the desired acceptance range.

3.2 QA DATA FROM PLANT B SITE SAMPLES

3.2.1 Volatile Organic Analytes

3.2.1.1 Precision Data--

Table L-25 presents the results of replicate analyses of organic waste sample No. 301 from the Plant B site. The precision for the analyses of most of the compounds is excellent, with RSDs of 2 to 12%. For benzene the precision found is near the limit of the desired acceptance range (RSD = 31%).

Table L-26 gives the results of replicate analyses of aqueous waste sample No. 302 for the Plant B site. The precision for the analyses of all the compounds is excellent (RSDs = 0 to 8%).

3.2.1.2 Accuracy Data--

Table L-27 presents the recoveries of selected volatile organic analytes from replicate spiked samples of organic waste No. 301 from the Plant B site. Trichloroethylene, benzene, and tetrachloroethylene and also the surrogate compounds D_4-1,2-dichloroethane and D_{10}-ethylbenzene gave good recoveries and the RSDs of the determinations are within the desired range. The recovery for carbon tetrachloride is low and outside the desired acceptance range and furthermore the precision for the carbon tetrachloride determination is rather poor (RSD = 48%). The precision of the chloroform determinations was acceptable but the recovery was low (56%).

Table L-28 presents the recoveries of volatile organic surrogate compounds from all samples from the Plant B site. Recoveries for D_{10}-ethylbenzene are all within the desired acceptance range. Recoveries of D_4-1,2-dichloroethane are within the desired acceptance range for all samples except the organic waste samples. Recoveries of this surrogate, which are high and outside the desired acceptance range, may have been caused by mass spectral interference from other components of the waste mixture. It is also possible that the very small amount of sample taken for analysis caused the analyte (surrogate) measurements to fall into a range where the response of the compound is nonlinear compared to the standard.

3.2.2 Semivolatile Organic Analytes

3.2.2.1 Precision Data--

Table L-29 presents the results of analyses of replicate extracts of organic waste sample No. 301 from the Plant B site. The results indicate excellent reproducibility with the 12 compounds quantified by comparison with an authentic standard showing RSDs from 4% to 20%, and seven of the eight compounds, which were quantified by using an estimated response factor, having RSDs from 1% to 29%. Caprolactam, detected in only one sample at a low level (30 μg/g) and not detected in the other two, may simply have been present at a level below the detection limit for this particular compound in two samples, or the presence of caprolactam in only one sample may indicate some slight inhomogeneity of the waste sample from which weighed portions were taken.

With respect to the overall excellent reproducibility of analysis, it is worthwhile to point out that all but two of the twenty compounds measured were present at moderate concentrations (50 to 3,000 μg/g). We have often observed during this and other projects that greater departures from good precision (and by extension from good accuracy) occur when attempting to measure very low or extremely high concentrations of analytes.

Table L-30 presents the results of analyses of replicate extracts of aqueous waste sample No. 302 from the Plant B site. The reproducibility of ten of the twenty determinations reported is good with RSDs of 4% to 21%. The reproducibility of the determinations of the other analytes is outside the desired acceptance range with RSDs of 34% to 173%. Inspection of the data lead to the conclusion that sample inhomogeneity was the principal cause of the poor reproducibility. The inhomogeneity was likely characterized by irregular distribution of solid particles with which some analytes were associated. Poor reproducibility for butylbenzylphthalate and bis(2-ethylhexyl)phthalate may have arisen from slight contamination of samples 302-B-2 with these two common environmental contaminants.

3.2.2.2 Accuracy Data--

Table L-31 gives the recoveries of selected semivolatile analytes from replicate samples of spiked organic waste No. 301 from the Plant B site. Most of the average recoveries are in the desired acceptance range but the average recovery for phenol is 180% and the average recovery for the surrogate compound D_{12}-chrysene is 46%. The reproducibility for this set of analyses was poor, with more than half the determinations showing RSDs over 30%. It is believed that the main reason for the great variations in the accuracy and precision of these results lies in the fact that due to a calculation error, the samples were spiked at an unsuitably low level: at one-fifth the level which would normally be chosen. Furthermore, inspection of the raw data showed that on the day these spiked samples were analyzed, the GC/MS system was producing more variable measurements on the internal standard than usual. These two problems taken together probably account for the inconsistent recovery data from these analyses.

Table L-32 presents the recoveries of selected semivolatile analytes from replicate spiked aqueous waste No. 302 from the Plant B site. The results are extremely variable depending on the compound used. Hexachlorobenzene and also the surrogate compounds decafluorobiphenyl, D_8-naphthalene and D_{12}-chrysene were all recovered very well with good precision. The recoveries for phenol (too high) and for 1,2,4-trichlorobenzene (too low) and naphthalene (too low) are outside the desired acceptance range. The main reason for the great variations in the accuracy and precision of these results again lies in the fact that, due to a calculation error, the samples were spiked at an unsuitably low level; at one-fifth the level which would normally be chosen. Furthermore, inspection of the raw data showed that on the day these spiked samples were analyzed the GC/MS system was producing more variable measurements on the internal standard than usual. These two problems taken together probably account for the inconsistent recovery data from these analyses.

Table L-33 gives the recoveries of selected semivolatile organic analytes from water samples spiked in triplicate. The precision of these determinations is good with only dimethylphthalate having an RSD slightly (high) outside the desired acceptance range. Recoveries, on the other hand, are fair to poor for most of the compounds studied. There is a marked trend for the more volatile compounds to have the lower recoveries and this may have arisen from volatility losses during sample preparation. The high recoveries for butylbenzylphthalate and bis(2-ethylhexyl)phthalate may be attributable to contamination with these two very common environmental contaminants.

Table L-34 presents the recoveries of selected semivolatile analytes from clean XAD resin samples spiked in triplicate. The precision of these determinations is quite good; all the RSDs lie within the desired acceptance range. Recoveries of the analytes ranged from 52% to 132% with eight of the twelve compounds having recoveries within or very near the desired acceptance range. No obvious explanation presents itself for the low recoveries of 1,2,4-trichlorobenzene and D_8-naphthalene. The rather high (132%) recovery of butylbenzylphthalate may be due to contamination with this common contaminant.

Table L-35 presents a summary of the recoveries of the semivolatile surrogate compounds for all samples from the Plant B incineration site. Recoveries of decafluorobiphenyl from spiked and unspiked waste samples range from 88 to 160% with the mean for the 20 analyses being 118%. On the whole the recoveries of decafluorobiphenyl from waste samples are high. This is believed to be due to mass spectral interference from other compounds in the waste samples. Recoveries of decafluorobiphenyl from the water and MM5 train samples range from 14% to 215% with a mean of 69%. The low recoveries of decafluorobiphenyl from some of these samples is probably due to volatility losses during sample preparation.

Recoveries of D_8-naphthalene generally parallel the recoveries of decafluorobiphenyl in similar samples; recoveries of D_8-naphthalene from all the waste samples averaged 124% with a range of 76 to 170%. The high recoveries are believed to be due to mass spectral interferences from other compounds in the waste samples. Recoveries of D_8-naphthalene from the water and MM5 train samples averaged 81% with a range of 33 to 135% with one unexplained anamalous value of 395%. Low recoveries of D_8-naphthalene from some of the water and MM5 train samples are probably due to volatility losses during sample preparation.

Recoveries of D_{12}-chrysene do not follow the same pattern as recoveries for decafluorobiphenyl and D_8-naphthalene. Recoveries from organic waste samples averaged only 28% and from aqueous waste 57%. Low recoveries of D_{12}-chrysene from organic waste feeds and similar complex mixtures have often been observed. The reasons are not clearly understood. Recovery of D_{12}-chrysene from water and MM5 train samples averaged 73% with a range of 26% to 195%. No pattern to the high and low recoveries is apparent.

3.2.3 Metals

3.2.3.1 Precision Data--

Table L-36 presents the results of analyses for metals in triplicate samples of organic waste No. 301 from the Plant B site. Of the elements detected all showed excellent analytical precision except for selenium which was detected in only one of the triplicate samples; this indicates relatively poor reproducibility for this element. Note that a more dilute aliquot (301-IC-4) of this sample was later analyzed in order to quantify calcium and phosphorous; the poor precision for the analytes during this analysis is likely due to their low concentration in the sample.

Table L-37 presents the results of analyses for metals in replicate samples of aqueous waste sample No. 302 from the Plant B site. All of the elements found were measured with excellent precision.

3.2.3.2 Accuracy Data--

Table L-38 presents the recovery data for metals spiked into organic waste No. 301 from the Plant B site. Two spiking experiments were carried out. In the first one, 11 elements (As, Ba, Be, Cd, Cr, Hg, Ni, Pb, Sb, Se, and Tl) were spiked at the 40-ppm level. Recoveries for most of the elements were within the desired acceptance range of 70 to 120% recovery. Recoveries for arsenic, mercury, and thallium were slightly too high (150%, 130%, and 140%, respectively). These results are probably due to two factors. The contribution to the measurement of an element in the spiked sample of amounts of element not detected in the unspiked sample increases the apparent recovery of that element. A second factor in high reported recoveries is the effect of frequently rounding numbers upward when three significant figures from measurement or calculation are rounded to two significant figures, e.g., 145 µg rounded to 150 µg; 105% rounded to 110%, etc. Measurement of selenium suffers severely from interference by calcium and the calculated determinations and recoveries for selenium in these samples probably has little validity.

In the second spiking experiment, barium, lead, chromium, and antimony were spiked at several hundred parts per million each. Recoveries for barium and antimony were acceptable; recoveries for chromium and lead were slightly below the desired acceptance range.

Table L-39 gives the recovery data for 12 elements spiked into aqueous waste No. 302 from the Plant B site. Recovery for 10 of the elements is very good. Silver was probably lost by silver chloride precipitation during the sample preparation. Measurement of selenium by ICP suffers from calcium interference, and in the presence of calcium in the sample, spiking and recovery data for selenium have little validity.

Table L-40 gives the recovery for beryllium spiked into ash sample No. 203 from the Plant B site. The recoveries for beryllium are acceptable. The samples were spiked prior to digestion according to the standard protocol. The sample, however, contained extremely high concentrations of common elements (calcium, iron, etc.) and for this reason the samples were analyzed as dilute solutions. Under these conditions recoveries of the spiked elements, except beryllium, could not be measured.

3.3 QA DATA FROM UPJOHN SITE SAMPLES

3.3.1 Volatile Organic Analytes

3.3.1.1 Precision Data--

Table L-41 presents the results of analysis of replicate tetraglyme extracts of liquid waste from the Upjohn site. For toluene the precision is good. For the other volatile POHCs found in the waste, chloroform, carbon tetrachloride, trichloroethylene, and benzene the precision is excellent.

3.3.1.2 Accuracy Data--

Table L-42 gives the recoveries of selected volatile organic analytes from spiked waste No. 301 from the Upjohn site. The selected POHCs, carbon tetrachloride, trichloroethylene, toluene, and chloroform show extraordinarily high recoveries, many times the expected values. The overall reproducibility of the measurements is not bad. In view of this fact and the excellent recovery and reproducibility shown by the surrogate D_{10}-ethylbenzene, it is possible that the POHCs being determined actually increased in the spiked aliquots relative to the unspiked samples. One mechanism for this to occur is via chemical reactions in the waste mixture. This particular organic waste is an extremely complex mixture of chlorine and nitrogen-containing compounds and on-going reactions in the mixture may be expected.

Table L-43 gives the recoveries of the volatile organic surrogate compounds for all samples analyzed for VOAs by purge and trap from the Upjohn site. The D_4-1,2-dichloroethane showed good recoveries for all but two samples; in water samples 205 and 305 the recoveries were (high) outside the desired acceptance range. Recoveries for D_{10}-ethylbenzene from the waste feed samples were quite low. The reason for this is not known. Possibly some compound(s) in the waste (chlorinated?) reacted with the D_{10}-ethylbenzene. Recovery of D_{10}-ethylbenzene from the three make-up water samples is good for two of the samples; recovery is (high) slightly outside the desired acceptance range for sample No. 205.

3.3.2 Semivolatile Organic Analytes

3.3.2.1 Precision Data--

The results of replicate analyses of the liquid waste feed are given in Table L-44. The precision of the results is very good for all compounds except for the surrogate compound D_{12}-chrysene, which are slightly outside the desired acceptance range.

Table L-45 presents the results of repeated analysis of a single sample (extract of front XAD plus condensate) derived from the MM5 train. Overall the precision of the analyses is very good (RSD ≦ 20%) and indicates the typical run-to-run repeatability generally obtained by GC/MS analysis. An exception to the overall good results was noted for naphthalene for which the third analysis yielded higher results than the first and second analyses.

3.3.2.2 Accuracy Data--

Table L-46 gives the recoveries of selected semivolatile POHCs spiked into triplicate samples of liquid waste No. 301 from the Upjohn site. Recoveries and precision are very good for 1,2,4-trichlorobenzene, naphthalene, 2,6-toluene diisocyanate and the surrogate compound D_8-naphthalene. Recoveries were zero for both bis-(2-ethylhexyl)phthalate and the surrogate compound D_{12}-chrysene. This is probable due to the fact that these compounds were too dilute to measure because the extracts were analyzed at 1/10 the original concentration in order to keep the dichlorobenzenes in a measurable range. The precision is good but the recovery is low for decafluorobiphenyl. The reason is not known. For m-dichlorobenzene the recovery was calculated to be zero for one of the triplicate samples and this caused the average recovery to be low and the precision to be very poor. A laboratory error may have been responsible for the anomalous zero recovery.

Table L-47 gives the recoveries of selected semivolatile POHC analytes from replicate spiked water. The precision of analyses for all the compounds is within the desired acceptance range. Recoveries for most of the compounds are good to excellent. Phenol (35% recovery) exhibits a low recovery from water because it is polar and acidic and is not efficiently extracted from neutral water into an organic solvent. Decafluorobiphenyl (55% recovery) may exhibit a rather low recovery due to volatility losses during concentration of the organic solvent (methylene chloride) used for the extraction. Toluene-2,6-diisocyanate exhibits zero recovery from water, probably due to its decomposing in aqueous solution.

Table L-48 gives the recoveries of selected semivolatile POHCs from triplicate samples of spiked XAD. Both the recovery and precision data are rather variable. Phenol and naphthalene show too-high recoveries, probably due to "bleed" of these two compounds from the XAD istelf. The same phenomenon gives rise to poor precision for these compounds during recovery experiments from XAD resin. Recovery for o-dichlorobenzene is somewhat outside the expected acceptance range (high) for unknown reasons. Recovery for the surrogate compound D_8-naphthalene is too high for unknown reasons. Variation in the recovery of D_{12}-chrysene gives rise to poor precision (RSD = 63%). It is not known if a phenomenon in the laboratory (e.g., absorption onto glassware) or GC/MS instrumental fluctuations caused the poor precision for the recovery of this compound.

Table L-49 gives the recoveries for the semivolatile surrogate compounds for all the samples from the Upjohn incineration test. Recoveries for decafluorobiphenyl range from very good (95%) to well below (24%, 28%) the desired acceptance range. Losses of decafluorobiphenyl during sample preparation are generally due to volatility losses. Recoveries for D_8-naphthalene are scattered below (38%), within, and above (170%) the desired acceptance range. Low recoveries are probably due to volatility losses during sample preparation. The two high values for D_8-naphthalene in spiked XAD may have arisen from interferences from background compounds falsely enhancing the signal for the characteristic D_8-naphthalene ion. Recoveries of D_{12}-chrysene from the Upjohn samples also range from low (0%, 10%) to high (190%). The low or zero recoveries from the unspiked and spiked waste feeds are due to the extract being diluted to the point where D_{12}-chrysene was not accurately measureable. The reasons for the high recovery from the water blank (190%) is not known. The reasons for the variability in the recovery of D_{12}-chrysene are not well understood.

3.4 QA DATA FROM ZAPATA SITE SAMPLES

3.4.1 Volatiles

3.4.1.1 Precision Data--

Table L-50 presents the results of analyses for volatile analytes in replicate extracts of liquid waste No. 201 from the Zapata site. The precision of the determinations for carbon tetrachloride, trichloroethylene, and chlorobenzene are excellent. The precision of the determinations for methylene chloride was somewhat outside the desired acceptance range. The reason for this is not known, but methylene chloride is a very volatile compound that can easily escape from a sample through volatility. The precision for the toluene determinations is poor due to a very high value being found for one of the three triplicate samples. The reason for this is not known.

3.4.1.2 Accuracy Data--

Table L-51 presents the recoveries of selected volatile analytes from triplicate spiked waste feed No. 201 from the Zapata site. The recoveries for the analytes carbon tetrachloride, trichloroethylene, chlorobenzene, and methylene chloride as well as the surrogate compound D_{10}-ethyl benzene are excellent and also the precision of these determinations are all within the desired acceptance range. Recovery of the surrogate D_4-1,2-dichloroethane was unexplainably low in all three replicates.

Table L-52 gives the recoveries of the volatile surrogates D_4-1,2-dichloroethane and D_{10}-ethylbenzene from the waste samples from the Zapata site. Recoveries for D_{10}-ethylbenzene are very good with only one value (130%) being (high) slightly outside the desired acceptance range. The recoveries of D_4-1,2-dichloroethane are good with only two values being (low) outside the desired acceptance range.

3.5 QA DATA FROM AMERICAN CYANAMID SITE SAMPLES

3.5.1 Volatile Organic Analytes

3.5.1.1 Precision Data--

Table L-53 presents the results of analyses of replicate extracts of waste feed No. 301 from the American Cyanamid site. All the volatile POHCs except toluene were present at concentrations below quantifiable levels so that their precision data is not very meaningful. The precision data for toluene are good.

3.5.1.2 Accuracy Data--

Table L-54 presents the recoveries of selected volatile POHCs spiked into waste feed No. 301 from the American Cyanamid site. Recoveries for all the analytes measured (carbon tetrachloride, trichloroethylene, chloroform, toluene) as well as the surrogates D_4-1,2-dichloroethane and D_{10}-ethylbenzene are excellent (range 85 to 120%) and the precision of the determinations is excellent. (Range of RSDs 1 to 10%.)

Table L-55 presents the recoveries of selected volatile analytes from replicate spikes of sample No. 401 from the American Cyanamid site. Recoveries and precision of the determinations for trichloroethylene, chloroform, benzene and the surrogate D_4-1,2-dichloroethane are excellent. The average recovery of carbon tetrachloride (58%) is (low) outside the desired acceptance range and shows a precision just within the acceptable range. The surrogate D_{10}-ethylbenzene shows a very low recovery (38%) and the precision (RSD = 41%) is outside the desired acceptance range.

Table L-56 gives the recoveries of the volatile surrogate compounds for all the waste feed samples from this incinerator site. Five of the seven samples showed very good recovery of D_4-1,2-dichloroethane and recoveries from the other two samples (130%, 130%) were only sightly (high) outside the desired acceptance range. D_{10}-ethylbenzene showed one good (80%) and one poor (53%) recovery in samples No. 101 and No. 201 respectively. It was not possible to determine D_{10}-ethylbenzene in the other samples because a coeluting compound caused saturation of the D_{10}-ethylbenzene peak and precluded its measurement.

3.5.2 Semivolatile Analytes

3.5.2.1 Precision Data--

Replicate extracts of waste samples from the site were not analyzed.

3.5.2.2 Accuracy Data--

Replicate spiked wastes were not analyzed owing to the composition of the wastes from the American Cyanamid site. Wastes No. 201, 301, and 501 each contained more than 50% aniline and more than 20% aminophenols and thus meaningful spiking experiments could not be done. Waste No. 401 contained more than 60% nitrobenzene and thus for the same reason meaningful spiking experiments could not be carried out.

Table L-57 presents the recoveries of selected semivolatile POHCs from spiked XAD resin. Aniline was very poorly recovered and may not be readily extracted from XAD resin. The precision for this compound was also very poor because aniline was not detected at all in two of the three spiked XAD samples. Recoveries for naphthalene and phenol were too high; naphthalene and phenol may "bleed" from XAD resin and that fact probably accounts for the high recoveries and rather poor precision of the naphthalene and phenol recoveries. For nitrobenzene, the recovery and precision fall within the desired acceptance range. The peak measured for 2-aminophenol was subject to interference by some unknown compound which led to calculation of very high recovery values which do not accurately reflect the recovery of the compound from the XAD. Recoveries of the surrogate compounds decafluorobiphenyl and D_8-naphthalene are within the desired acceptance range. Recovery of D_{12}-chrysene is lower than the desired acceptance range but the precision of measurement is good.

Table L-58 presents the recoveries of selected semivolatile POHC analytes from spiked water. All the precision measurements are very good. The recovery of phenol is low. Phenol is polar and acidic and is poorly extracted from neutral water. The calculated recovery of 2-aminophenol is too high. We attribute this to probable instrumental limitations in measuring the response of the compound in the calibration standard. Orthoaminophenol (2-aminophenol) gives a very low response in the GC/MS system and very low responses cannot be measured with good accuracy. Recoveries of the remaining spiked analytes are very good.

Table L-59 presents the recoveries of the semivolatile surrogate compounds from all samples from the American Cyanamid site. Decafluorobiphenyl and D_8-naphthalene were very well recovered from the waste samples and from the XAD and spiked water samples; one exception is XAD sample No. 904 from which apparent decafluorobiphenyl recovery is zero. Possibly this compound was accidentally not added to this sample. Recoveries of decafluorobiphenyl and D_8-naphthalene from the spiked XAD samples are a little high, probably owing to interference from uncharacterized compounds "bleeding" from the XAD. Recoveries of D_{12}-chrysene from most of the samples are within the desired acceptance range. The reason for low recoveries from five different XAD samples is not known.

3.5.3 Metals

3.5.3.1 Precision Data--

Table L-60 gives the results of analyses of replicate samples of liquid waste No. 301. Most of the precision data are very good. Four elements, boron, magnesium, phosphorus, and zinc, have RSDs higher than the 30% acceptance level. The reason for this is not known, but these are common elements and the data may reflect some slight contamination of the analytical samples. The actual sample levels measured were low, and that means that any contamination or other variation in the measurement has a pronounced effect on the calculated precision.

3.5.3.2 Accuracy Data--

Table L-61 gives the recovery data for duplicate spiked samples of liquid waste No. 301 from the American Cyanamid site. Recoveries of most of the spiked elements are very good. A few elements show low recoveries. Silver often gives low recoveries when spiked into chlorine-containing organic wastes as it readily precipitates out as silver chloride during the sample preparation. The reasons for the low recoveries of barium, antimony, and zinc are not known.

3.6 QA DATA FROM MITCHELL SITE SAMPLES

3.6.1 Volatile Organic Analytes

3.6.1.1 Precision Data--

Table L-62 presents the results of analysis of replicate extracts of organic waste No. 401. Few volatile POHCs were found and these were at low concentrations. Chloroform and tetrachloroethylene were found at such low levels that precision data are not applicable. The precision for quantification of toluene and 1,1,1-trichloroethane is very good. Precision for methylene chloride is poor (RSD = 68%). The reason for this is not known, but methylene chloride is a volatile compound which can readily vaporize from aqueous samples.

Table L-63 presents the results of analysis of replicate extracts of organic waste No. 402. Precision is excellent for the five POHCs measurable in this set of samples. The data in this table are a good example of the general trend that analytes can be measured with greater accuracy and precision at levels well above their detection limits and below the level at which ion peaks become saturated.

3.6.1.2 Accuracy Data--

Table L-64 gives the recoveries of volatile organic analytes from spiked aqueous waste No. 401. All the recoveries are less than optimal and the recoveries for carbon tetrachloride, trichloroethylene, and toluene are extraordinarily low. In view of the good precision, it seems likely that the sample itself may have caused the low recoveries, perhaps through chemical reaction.

Table L-65 gives the recoveries of volatile organic analytes from spiked organic waste No. 402. The average recovery for carbon tetrachloride is outside the desired acceptance range, and the precision of the recoveries is also poor. Recovery of tetrachloroethylene is low, while the reproducibility is within the acceptance range. The recovery of trichloroethylene is excessively high but the precision of the measurements is very good. Benzene and the two surrogate compounds were well recovered, with good precision.

Table L-66 gives the recoveries of the volatile organic surrogate compounds from all Mitchell samples. Recoveries of D_4-1,2-dichloroethane from organic waste are variable, with four of the six samples showing recoveries within the desired acceptance range. Recoveries of D_{10}-ethylbenzene were slightly better, with only one out of six samples giving an excessively low recovery. Recoveries of D_4-1,2-dichloroethane from the aqueous waste were excessively high, while recoveries of D_{10}-ethylbenzene were too low. Aliquots of the aqueous waste tetraglyme extract taken for analysis were much larger than usual, and this led to very high area counts for the ion peaks of the surrogate compounds. Measurements were thus made well outside the range of linear response for both compounds, and this probably is the cause of the recoveries of the surrogate compounds outside the desired acceptance range.

3.6.2 Semivolatile Compounds

3.6.2.1 Precision Data--

Replicate extracts of the waste samples were not analyzed.

3.6.2.2 Accuracy Data--

Spiked waste samples were not analyzed. The aqueous waste from this site contained phenol at a high level (30,000 to 40,000 ppm); meaningful spiking experiments cannot be carried out on such concentrated analytes. The organic waste contained small amounts of nonhalogenated POHCs. It was not possible to carry out recovery experiments on this waste owing to time, personnel, and instrumental limitations.

Table L-67 gives the recoveries of selected semivolatile POHCs from spiked water. The precision of the determinations is excellent. The recoveries are excellent for all compounds except phenol (48% recovery), which is acidic and polar and is not efficiently extracted from neutral water.

Table L-68 gives the recoveries of selected semivolatile POHCs from spiked XAD. Most of the recoveries lie high and outside the desired acceptance range. The high recoveries and poor precision data for phenol and naphthalene may be accounted for by the XAD itself "bleeding" these compounds into the extracting solvent; phenol and naphthalene are nearly always found in XAD blanks. However, the overly high results from the other compounds are not readily explained. The problem of low recovery and poor precision of D_{12}-chrysene has frequently been observed in analyzing for this particular surrogate compound.

Table L-69 gives the recoveries for the semivolatile surrogate compounds from all samples. Recovery of decafluorobiphenyl from the waste samples is good overall, except for 12% recovery in aqueous waste sample No. 101. The low recovery values for D_{12} chrysene in the liquid waste are often observed and are probably due to little-understood internal phenomena of the GC/MS instrument or sample matrix effects.

3.7 QA DATA FROM DU PONT SITE SAMPLES

3.7.1 Volatile Organic Analytes

3.7.1.1 Precision Data for Volatile Organic Analytes--

Table L-70 presents the results of analyses of triplicate tetraglyme extracts of liquid waste No. 202. The precision for most of these analyses was excellent (RSD $\leqq$ 20%). The precision for 3,4-dichloro-1-butene is good (RSD =30%). The chemical reactivity of the dichlorobutene or slight sample inhomogeneity could account for the variation in the measurements for this compound.

3.7.1.2 Accuracy Data--

Table L-71 gives the recoveries of volatile organic analytes from spiked liquid waste No. 202. Carbon tetrachloride, trichloroethylene, methylene chloride, and the surrogate compounds D_4-1,2-dichloroethane and

D_{10}-ethylbenzene were all very well recovered (77-100% recovery) with very good precision. The recovery for chloroform is high and outside the desired acceptance range, but the precision is good. The cis-1,4-dichloro-2-butene gave calculated recoveries of zero in the spiked samples, probably due to reaction of the added compound with other components of the waste mixture. (Also see results of spiking semivolatile compounds, below.)

Table L-72 presents the recoveries of the volatile surrogates, D_4-1,2-dichloroethane and D_{10}-ethylbenzene, from the samples from the Du Pont site. Recoveries of both surrogates from water samples are excellent. Recoveries of both surrogates from drum wastes, when measurable, are excellent. For some samples of drum wastes and liquid waste, the aliquots taken for analysis for volatiles were so small that D_4-1,2-dichloroethane was diluted to below its detection limit. Recoveries for D_{10}-ethylbenzene from liquid waste were quite variable, possibly due to the very reactive sample components.

3.7.2 Semivolatile Organic Analytes

3.7.2.1 Precision Data--

Replicate extracts of unspiked liquid waste were not analyzed.

3.7.2.2 Accuracy Data--

Table L-73 presents the results of analyses for semivolatile POHCs spiked into triplicate samples of waste No. 202. The average recoveries are variable in the extreme, while most of the precision data are fair to good. From this and the fact that the native concentration used for recovery calculations was obtained from a single analysis, the more obvious explanation is that the unspiked sample may not have been homogeneous and the analytical results from it do not accurately reflect the amounts of POHCs present in a well-homogenized sample. An alternative explanation is that the liquid waste is chemically reactive and that, during the time interval between the analysis of the unspiked and spiked samples, chemical reactions took place which formed some POHCs and decomposed others. In view of the large amounts of chemically reactive dichlorobutenes present in the liquid waste, this scenario seems quite probable. (Compare the results from spiking in volatile POHCs, Table L-71.)

Table L-74 gives the recoveries of selected POHCs and semivolatile surrogate compounds from spiked water. The precision of almost all compounds except 3,4-dichloro-1-butene is excellent; the results for 3,4-dichloro-1-butene are still within the desired acceptance range. The recoveries for 3,4-dichloro-1-butene and phenol are low (each 28%). Phenol is an acidic polar compound, not readily recovered from neutral pH water. The low recovery of 3,4-dichloro-1-butene is attributed to loss during extraction and concentration due to its volatility. Recoveries of the other selected analytes typically fall slightly below the desired acceptance range of 70% recovery.

Table L-75 presents the results of analyses of XAD resin fortified in triplicate. Recoveries range from 38 to 121%, averaging 61%, slightly below the desired acceptance range. p-Chlorophenol and methylbenzyl chloride (all isomers) show recoveries of 38% and 41%, respectively. No reason is known for low recoveries of these two compounds. Precision of the data is very good for most compounds in this group, indicating that the factors influencing the recoveries operated equally on all three samples. Exceptions are o-chlorotoluene (RSD = 38%) and p-chlorophenol (RSD = 38%). No reason is known for the variability of recovery of these two compounds.

Table L-76 gives the recovery of the semivolatile surrogate compounds from all samples from the DuPont site. Most of the recoveries, except those from the waste samples, fall in the range of 70 to 120%, within the desired acceptance range. The results for sample No. 130, the XAD field blank, are at variance with the results from the other XAD samples for unknown reasons. Recoveries of decafluorobiphenyl and D_8-naphthalene from the waste samples average 150% and 190%, respectively, outside the acceptance range. It is believed that little-understood effects of the complex and reactive sample matrix may have caused the apparent high recoveries. The D_{12}-chrysene (average recovery from wastes, 68%) is apparently not affected by the sample matrix effects.

3.7.3 Metals

3.7.3.1 Precision Data--

Table L-77 presents the results of replicate analyses of paint waste composite No. 501 from the Du Pont site for metals. The precision of analysis for all the metals reported is somewhat outside the desired acceptance range. The scatter of high and low values in the table suggests that the sample from which the replicates were taken may not have been homogeneous.

3.7.3.2 Accuracy Data--

Table L-78 presents recovery data for metals spiked into paint waste composite No. 501. The recoveries are excellent for all elements except boron, whose recoveries are slightly high. Precision of the analyses is very good for all the elements determined.

3.8 QA DATA FROM TWI SITE SAMPLES

3.8.1 Volatiles

3.8.1.1 Precision Data--

Table L-79 presents the results of triplicate analyses for volatile POHCs in extracts of organic waste No. 301. In order to obtain a quantifiable toluene response, replicate No. 1 was analyzed at one-tenth the concentration of replicates No. 2 and 3. This dilution accounts for the nondetection of chloroform, 1,1,1-trichloroethane, and tetrachloroethylene in replicate No. 1 while they are at measurable levels in replicates No. 2 and 3. The difference in the concentrations analyzed also accounts for the variation in the measured benzene concentrations. For most of the compounds present at moderate concentrations (carbon tetrachloride, trichloroethylene, benzene, and chlorobenzene) the RSDs are good to excellent. The reason for the variability of the methylene chloride results is not known.

Table L-30 presents the results of triplicate analyses for volatile POHCs in extracts of aqueous waste No. 308. The precision for most of the analytes is excellent. The greater variation in the results for chloroform and benzene may occur because accuracy (and therefore reproducibility) of the analyses are worse when measuring very low levels of an analyte.

3.8.1.2 Accuracy Data--

Table L-81 presents the recoveries of selected volatile organic analytes spiked into triplicate samples of organic waste No. 301 from the TWI site. The recoveries for carbon tetrachloride, trichloroethylene, chlorobenzene and the surrogate compound D_{10}-ethylbenzene are good and the reproducibility is very good. The average recovery for chloroform is within the desired acceptance range; the precision is outside the desired acceptance range. Methylene bromide was determined with good precision but the average measured recovery is (high) outside the desired acceptance range. This is probably due to some interfering component of the sample mixture contributing to the measurement of the intensity of the characteristic ion peak. The surrogate D_4-1,2-dichloroethane was not detected because it was diluted to below a detectable level when a very small aliquot of sample was taken for analysis.

Table 82 presents the recovery data for volatile POHCs spiked into triplicate samples of aqueous waste No. 308 from the TWI site. All the recoveries are (low) outside the desired acceptance range. The recoveries for carbon tetrachloride, chloroform, and the surrogate compound D_4-1,2-dichloroethane are especially low. Generally good precision indicates that the same factors influenced all the samples in the same way. The data from this experiment indicate that some constituent of the waste itself may have reacted with the spiked analytes. Methylene bromide is a possible reactant, but the reactive constituent(s) of the aqueous waste may be compounds which were not identified during the analyses.

Table L-83 presents the recoveries of volatile surrogate compounds D_4-1,2-dichloroethane and D_{10}-ethylbenzene from the TWI samples. Recoveries of both surrogates from all the water samples was excellent. Recoveries of D_4-1,2-dichloroethane from the rest of the samples were generally very good, with the exception of a few low recoveries from organic waste samples. These low recoveries were doubtlessly due to the excessive diminution of the surrogate concentration as a result of taking small (and nonidentical) aliquots of the sample for the purge-and-trap analysis. Most of the recoveries for D_{10}-ethylbenzene are good. A few recoveries from the organic waste are too high, probably due to interfering co-eluting compounds. In two samples of the solid waste composite the D_{10}-ethylbenzene was "diluted out" due to the very small aliquot of sample taken for analysis.

3.8.2 Semivolatile Compounds

3.8.2.1 Precision Data--

Table L-84 presents the results of analysis for semivolatile POHCs in triplicate extracts of liquid organic waste No. 301. The precision is well within acceptance criteria for all of the three semivolatile POHCs found in the waste composite.

Table L-85 gives the results of analysis for semivolatile POHCs in triplicate extracts of solid waste composite No. 410. With the exception of di-n-butyl phthalate, a nearly ubiquitous environmental contaminant, the precision for the semivolatile POHCs found in this sample are very good.

3.8.2.2 Accuracy Data--

Table L-86 presents the recoveries of selected semivolatile POHCs spiked into replicate samples of solid waste composite No. 410. Six of the spiked analytes show high recoveries, nearly 200%, while six others show low recoveries, near 50%. The apparent low average recovery (37%) for hexachlorocyclopentadiene may be caused by this compound's known instability. D_{12}-Chrysene and bis(2-ethylhexyl) phthalate show rather low recoveries (40% and 53%, respectively). Rather low recoveries for these two compounds are frequently observed in similar analyses. There appears to be a correlation between the long retention time of D_{12}-chrysene and bis(2-ethylhexyl) phthalate and their low response, but the exact cause of this phenomenon has not been well explained. Several compounds show high recoveries (about 200%). The reason for this is believed to be related to the high levels of spiked hexachlorobutadiene and chlordane, which may have caused unexpected interferences with the detection of other compounds with the mass spectrometer detector. The good overall precision for the triplicate analyses indicates that the same causes for too high or too low recoveries were operating on all the spiked samples equally.

Table L-87 presents the results of analyses of replicate fortified XAD samples. The average recoveries are all excellent with the exception of hexachlorocyclopentadiene. The reason for this low recovery is probably the compound's poor stability. The precision for the replicate recoveries for all compounds except hexachlorocyclopentadiene is excellent. Precision for the hexachlorocyclopentadiene results (RSD = 35%) is not as good as for the other analytes.

Table L-88 gives the recoveries of the semivolatile surrogate compounds for all samples from the TWI site. Recoveries of decafluorobiphenyl and D_8-naphthalene range from 48 to 110% for most samples and are greater than 100% for the samples of wastes Nos. 410 and 462, which contained about 2% hexachlorocyclopentadiene. This compound may have affected the MS response of other compounds. Decafluorobiphenyl and D_8-naphthalene also show high recoveries from the chamber ash samples. The reason is not known. Recoveries for D_{12}-chrysene are consistently lower than for decafluorobiphenyl and D_8-naphthalene. This same phenomenon has been observed for many other sets of analyses as described above.

3.8.3 Metal Analytes

3.8.3.1 Precision Data--

Table L-89 gives the results of replicate analyses for metals in liquid organic waste No. 401 from the TWI site. The analytical precision for all the elements is excellent.

Table L-90 gives the results of replicate analyses for metals in solid waste composite No. 410 from the TWI site. The precision for most of the elements is within the desired acceptance range. The determinations for copper, calcium, and yttrium showed slightly more variation than the determinations for the other elements.

3.8.3.2 Accuracy Data--

Table L-91 gives the recovery data for elements spiked into replicate samples of liquid organic waste No. 401 from the TWI site. Recoveries for half of the spiked elements are within the desired acceptance range. Recoveries for the other spiked elements are somewhat high and outside the desired acceptance range.

Table L-92 gives the recovery data for metals spiked into solid waste composite No. 410 from the TWI site. Ten of the 16 elements spiked have recoveries within the desired acceptance range. The other six elements spiked show recoveries higher than the desired acceptance range.

TABLE L-1. VOLATILE ORGANIC COMPOUND PRECISION DATA (% RSD)

	Matrix			
	Aqueous waste		Organic/drum waste	
Compound	No. of measurements	Within desired range[a]	No. of measurements	Within desired range[a]
D_4-1,2-Dichloroethane (surrogate)	5	80%	15	87%
D_{10}-Ethylbenzene (surrogate)	5	100%	15	93%
Toluene	5	80%	10	70%
Methylene chloride	3	67%	7	71%
Methyl ethyl ketone (MEK)	1	100%	2	100%
Carbon tetrachloride	3	100%	15	87%
Trichloroethylene	4	100%	16	100%
Tetrachloroethylene	4	100%	8	100%
1,1,1-Trichloroethane	1	100%	1	100%
Chloroform	5	80%	12	92%
Benzene	3	67%	9	78%
Chlorobenzene	0	-	4	100%
cis-1,4-Dichloro-2-butene	0	-	1	100%
3,4-Dichloro-1-butene	0	-	1	100%
Bromochloromethane	1	100%	0	-
Methylene bromide	1	100%	1	100%

[a] % RSD of measurement ≦ 30%.

TABLE L-2. VOLATILE ORGANIC COMPOUND ACCURACY DATA (PERCENT RECOVERY)

Compound	Matrix					
	Water		Aqueous waste		Organic and drum waste	
	No. of measurements	Within desired range[a]	No. of measurements	Within desired range[a]	No. of measurements	Within desired range[a]
d_4-1,2-Dichloroethane (surrogate)	37	97%	34	62%	84	70%
d_{10}-Ethylbenzene (surrogate)	37	97%	34	53%	84	75%
Toluene	0	-	7	43%	10	40%
Carbon tetrachloride	0	-	9	33%	28	64%
Trichloroethylene	0	-	10	40%	28	57%
Chloroform	0	-	10	40%	28	39%
Benzene	0	-	4	25%	13	69%
Chlorobenzene	0	-	0	-	6	100%
cis-1,4-Dichloro-2-butene	0	-	0	-	3	0%
Methylene bromide	0	-	3	0%	3	33%
Tetrachloroethylene	0	-	7	43%	10	70%
Methylene chloride	0	-	0	-	6	83%
	74		118		303	

[a] Percent recovery = 70 to 130%.

TABLE L-3. SEMIVOLATILE ORGANIC COMPOUND PRECISION DATA (% RSD)

	Matrix							
	Aqueous waste		Organic/drum waste		Water[a]		XAD[a]	
Compound	No. of measurements	Within desired range[b]	No. of measurements	Within desired range[b]	No. of measurements	Within desired range[b]	No. of measurements	Within desired range[b]
Decafluorobiphenyl (surrogate)	4	75%	7	100%	6	83%	7	86%
D_8-naphthalene (surrogate)	4	75%	7	100%	6	100%	7	86%
D_{12}-chrysene (surrogate)	4	50%	7	57%	6	100%	7	43%
C_{13}-phenol (surrogate)	0	-	0	-	0	-	7	43%
Phenol	4	75%	4	75%	5	80%	1	100%
Isophorone	1	100%	2	100%	2	100%	6	33%
2,4-Dimethylphenol	2	0%	2	50%	2	100%	1	0%
Naphthalene	3	67%	8	63%	2	100%	2	50%
bis(2-Ethylhexyl)phthalate	1	0%	4	75%	5	100%	7	43%
Butylbenzylphthalate	0	-	1	100%	2	100%	3	67%
N,N-dimethylacetamide	2	50%	1	100%	1	0%	0	-
Dimethylphthalate	2	100%	2	100%	2	50%	1	100%
1,2,4-Trichlorobenzene	2	100%	6	83%	3	100%	2	50%
Diethylphthalate	1	100%	1	100%	3	100%	4	75%
Hexachlorobenzene	1	100%	3	67%	2	100%	3	67%
Dodecanol	1	100%	1	100%	1	100%	3	67%
Butylbenzylphthalate	1	0%	1	100%	1	100%	1	100%
Ethylhexylchloride	1	0%	1	100%	0	-	1	100%
Diphenyl ether	1	0%	1	100%	0	-	0	-
Octachlorocyclopentene	0	-	1	100%	0	-	0	-
Isodrin	0	-	1	100%	0	-	0	-
m-Dichlorobenzene	0	-	1	100%	0	-	0	-
p-Dichlorobenzene	0	-	1	100%	1	100%	1	0%
o-Dichlorobenzene	0	-	1	100%	1	100%	1	100%
Phenyl isocyanate	0	-	1	100%	1	100%	1	100%
Aniline	0	-	1	100%	0	-	0	-
2,6-Toluene diisocyanate	0	-	1	100%	1	100%	1	0%
Nitrobenzene	0	-	1	100%	1	0%	1	100%
2-Aminophenol	0	-	0	-	1	100%	1	100%
o-Chlorotoluene	0	-	0	-	1	100%	1	100%
Benzylchloride	0	-	1	0%	1	100%	1	0%
Hexachloroethane	0	-	1	100%	1	100%	1	100%
1,2,3,4-Tetrachlorobutane	0	-	1	100%	0	-	1	100%
p-Chlorophenol	0	-	1	100%	1	100%	1	100%
Methylbenzylchloride	0	-	1	0%	1	100%	1	0%
3,4-Dichloro-1-butene	0	-	1	100%	1	100%	1	100%
1,4-Dichlorobutane	0	-	0	-	1	100%	1	100%
trans-1,4-Dichloro-2-butene	0	-	0	-	1	100%	1	100%
Hexachloroethane	0	-	0	-	1	100%	1	100%
Hexachlorobutadiene	0	-	0	-	1	100%	0	-
Hexachlorocyclopentadiene	0	-	2	100%	0	-	1	100%
Octachlorocyclopentene	0	-	2	50%	0	-	1	0%
Di-*n*-butylphthalate	0	-	2	100%	0	-	1	100%
Chlordane	0	-	2	50%	0	-	1	100%
			1	100%	0	-	1	100%
	35		84		65		77	

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a Clean sample matrix for POHCs other than surrogates.

b % RSD ≤ 30% for triplicate measurements.

TABLE L-4. RESULTS OF REPEATED SEQUENTIAL GC/MS ANALYSIS OF A SINGLE SAMPLE (3042B1) OF ORGANIC WASTE (ROSS)

Compound	μg/g				% RSD
	Rep 1	Rep 2	Rep 3	Average	
N,N-dimethylacetamide	ND	ND	ND	ND	0
Phenol	2,600	3,000	2,100	2,600	18
Isophorone	18,000	24,000	14,000	19,000	27
Decafluorobiphenyl	300	410	240	320	27
2,4-Dimethylphenol	1,200	1,600	970	1,300	25
1,2,4-Trichlorobenzene	ND	ND	ND	ND	0
D_8-Naphthalene	340	470	280	360	27
Naphthalene	870	1,200	680	630	29
Dimethylphthalate	37	40	30	36	14
Diethylphthalate	24	30	20	25	20
Hexachlorobenzene	ND	ND	ND	ND	0
D_{12}-Chrysene	70	43	160	92	69
Bis(2-ethylhexyl)phthalate	170	120	450	250	72

TABLE L-5. RESULTS OF TRIPLICATE UNINTERRUPTED SEQUENTIAL ANALYSIS OF A SINGLE EXTRACT OF ORGANIC WASTE NO. 301-2B2 FOR SEMIVOLATILE POHCs

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD	Original analysis 301-2B2	Average for four results	% RSD for four results
Phenol	2,300	2,200	2,500	2,300	7	1,800	2,200	13
Decafluorobiphenyl	350	340	400	360	9	320	350	10
1,2,4-Trichlorobenzene	76	76	82	78	4	77	78	4
D_8-Naphthalene	190	190	200	190	3	330	230	30
Naphthalene	380	370	400	380	4	430	400	7
Dimethylphthalate	96	100	100	99	2	120	100	10
Dodecanol	730	680	700	700	4	680	700	3
Diethylphthalate	1,600	1,600	1,800	1,700	7	1,300	1,600	13
Hexachlorobenzene	32	31	35	33	6	28	32	9
Butylbenzylphthalate	1,200	1,600	900	1,200	28	490	1,000	45
D_{12}-Chrysene	34	45	25	35	29	68	43	43
Bis(2-ethylhexyl)phthalate	120	150	97	120	22	91	110	23

TABLE L-6. SEMIVOLATILE ORGANIC COMPOUND ACCURACY DATA (PERCENT RECOVERY)

	Matrix							
	Aqueous waste		Organic/drum waste		Water[a]		XAD[a]	
Compound	No. of measurements	Within desired range	No. of measurements	Within desired range	No. of measurements	Within desired range	No. of measurements	Within desired range
Decafluorobiphenyl (surrogate)	21	76%	71	48%	42	33%	66	39%
D_8-naphthalene (surrogate)	21	52%	71	30%	42	80%	54	41%
D_{12}-chrysene (surrogate)	14	36%	64	37%	27	93%	54	41%
C_{13}-phenol (surrogate)	0	-	0	-	0	-	3	0%
N,N-dimethylacetamide	3	0%	3	0%	2	0%	2	0%
Phenol	6	0%	9	0%	17	0%	16	25%
2,4-Dimethylphenol	3	0%	3	0%	5	100%	5	40%
Naphthalene	6	50%	15	40%	17	82%	20	40%
Dimethylphthalate	3	100%	3	0%	5	60%	5	100%
1,2,4-Trichlorobenzene	6	50%	12	58%	8	63%	11	73%
Isophorone	0	-	0	-	2	100%	2	100%
Diethylphthalate	0	-	0	-	8	88%	8	75%
Hexachlorobenzene	3	100%	6	17%	5	100%	8	100%
bis(2-Ethylhexyl)phthalate	0	-	6	0%	5	60%	8	100%
Dodecanol	0	-	0	-	3	33%	3	100%
Butylbenzylphthalate	0	-	0	-	3	33%	3	33%
m-Dichlorobenzene	0	-	3	33%	3	67%	3	67%
2,6-Toluene diisocyanate	0	-	3	100%	3	0%	3	100%
o-Dichlorobenzene	0	-	0	-	3	100%	3	0%
p-Dichlorobenzene	0	-	0	-	3	67%	3	100%
Aniline	0	-	0	-	3	67%	3	0%
Nitrobenzene	0	-	0	-	3	100%	3	67%
2-Aminophenol	0	-	0	-	3	0%	2	0%
Benzylchloride	0	-	3	0%	3	67%	3	0%
1,2,3,4-Tetrachlorobutane	0	-	3	67%	3	0%	3	0%
Methylbenzylchloride	0	-	0	-	3	0%	3	0%
Hexachloroethane	0	-	3	0%	3	67%	3	0%
3,4-Dichloro-1-butene	0	-	0	-	3	0%	3	0%
1,4-Dichlorobutane	0	-	0	-	3	33%	3	33%
trans-1,4-Dichloro-2-butene	0	-	0	-	3	0%	3	0%
o-Chlorotoluene	0	-	0	-	3	67%	3	67%
p-Chlorophenol	0	-	0	-	2	0%	3	0%
Hexachlorobutadiene	0	-	3	0%	0	-	3	100%
Hexachlorocyclopentadiene	0	-	3	0%	0	-	3	0%
Octachlorocyclopentene	0	-	3	0%	0	-	3	100%
Chlordane	0	-	3	0%	0	-	3	33%
Di-n-butylphthalate	0	-	3	33%	0	-	3	100%

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a Clean sample matrix for POHCs other than surrogates.

TABLE L-7. EPA METHOD CHECK ANALYSIS ACCEPTANCE CRITERIA FOR PERCENT RECOVERY INTO SPIKED SAMPLES

Compound	Low limit	High limit
1,2,3-Trichlorobenzene	50	200
Hexachlorobenzene	50	200
Hexachloroethane	50	175
o-, *m*-, and *p*-Dichlorobenzene	15	200
Hexachlorobutadiene	20	150
Isophorone	25	300
Naphthalene	50	160
Nitrobenzene	35	260
Bis(2-ethylhexyl)phthalate	50	400
Butylbenzene phthalate	40	200
Di-*n*-octylphthalate	50	300
Diethyl phthalate	20	200
Di-*n*-butylphthalate	50	180
Dimethyl phthalate	30	200
Chrysene	50	120
2,4-Dimethylphenol	3	140
Phenol	50	200

TABLE L-8. RESULTS OF ANALYSIS OF REPLICATE EXTRACTS OF AQUEOUS WASTE NO. 303 FROM ROSS SITE FOR VOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Toluene	21	31	23	25	21
Methylene chloride	24	24	24	24	0
Methyl ethyl ketone	240	320	320	290	16

TABLE L-9. RESULTS OF ANALYSIS OF REPLICATE EXTRACTS OF ORGANIC WASTE NO. 304 FROM ROSS SITE FOR VOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Carbon tetrachloride	5,200	6,300	5,500	5,700	10
Trichloroethylene	8,000	7,400	7,900	7,800	4.1
Tetrachloroethylene	8,200	7,600	8,500	8,100	5.6
Toluene	47,000	46,000	52,000	48,000	6.6
Methyl ethyl ketone	17,000	17,000	17,000	17,000	0
1,1,1-Trichloroethane	42,000	47,000	25,000	38,000	30

TABLE L-10. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF AQUEOUS WASTE NO. 303 FROM ROSS SITE

Compound	Found in[a] unspiked sample µg/g	Amount added µg/g	% Recovery				
			Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Carbon tetrachloride	ND	15	97	100	96	98	2.1
Trichloroethylene	ND	10	100	110	100	100	5.6
Chloroform	ND	10	96	110	98	100	7.5
Benzene	ND	2	150	170	160	160	6.3
Tetrachloroethylene	ND	5	120	140	83	110	25
Toluene	25	60	120	120	45	95	46
D_4-1,2-Dichloroethane	-	250	180	200	100	160	33
D_{10}-Ethylbenzene	-	250	140	170	97	140	27

[a] Average of three replicates.

TABLE L-11. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 304 FROM ROSS SITE

Compound	Found in[a] unspiked sample µg/g	Amount added µg/g	% Recovery				
			Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Carbon tetrachloride	5,700	11,600	92	90	88	90	2.2
Trichloroethylene	7,800	19,800	90	120	87	99	18
Chloroform	ND	1,000	120	140	110	120	12
Benzene	ND	520	120	150	130	130	11
Tetrachloroethylene	8,100	15,800	95	81	88	88	8.0
Toluene	48,000	94,700	62	54	56	57	7.3
D_4-1,2-Dichloroethane	-	250	170	170	150	160	7.1
D_{10}-Ethylbenzene	-	250	300	240	200	250	20

[a] Average of three replicates.

TABLE L-12. RECOVERIES OF VOLATILE SURROGATE COMPOUNDS IN ROSS SAMPLES (% RECOVERY)

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Pond waters and effluents	108	88	100
	208	120	100
	308	92	97
	109	95	110
	209	94	110
	309	82	110
	110	96	100
	210	110	100
	310	110	100
Aqueous waste	103	78	80
	203	74	72
	303-1	80	69
	303-2	87	80
	303-3	88	83
Organic waste	104	ND	110
	204	ND	110
	304-1	ND	140
	304-2	ND	110
	304-3	ND	90
Drum waste composites	510	110	110
	511	99	110
	512	91	95
	513	ND	100
	514	ND	100
	515	ND	94

ND = Not detected due to small amount of sample analyzed.

TABLE L-13. RESULTS OF ANALYSIS OF REPLICATE EXTRACTS OF ORGANIC WASTE NO. 304 FROM ROSS SITE FOR SEMIVOLATILE POHCs (µg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Phenol[a]	PNQ[c]	PNQ[c]	4,100	4,100	NA[d]
Isophorone[a]	28,000	28,000	30,000	29,000	4
2,4-Dimethylphenol[a]	1,700	2,300	PNQ[c]	2,000	21[e]
Naphthalene[a]	430	480	570	490	14
Bis(2-ethylhexyl)-phthalate	360	670	370	470	38
Butylbenzylphthalate[b]	100	140	140	130	18
Decafluorobiphenyl[a]	180	190	170	180	6
D_8-Naphthalene[a]	160	160	220	180	19
D_{12}-Chrysene[a]	180	180	180	180	0

[a] Quantified by comparison with an authentic standard.

[b] Quantified using an estimated response factor.

[c] PNQ = present but not quantifiable due to interferences.

[d] NA = not applicable.

[e] Based on only two values.

TABLE L-14. RESULTS OF ANALYSIS OF REPLICATE EXTRACTS OF AQUEOUS WASTE NO. 303 FROM ROSS SITE FOR SEMIVOLATILE POHCs (μg/g)

Compounds	Rep 1	Rep 2	Rep 3	Average	% RSD
N,N-dimethylacetamide	75	14	11	33	100
Phenol	150	200	73	141	45
Isophorone	10	13	12	12	13
2,4-Dimethylphenol	1	1	ND	0.7	87
Naphthalene	1	1	1	1	0
Decafluorobiphenyl	190	86	120	130	40
D_8-Naphthalene	180	56	53	96	75
D_{12}-Chrysene	170	230	240	210	18

TABLE L-15. RECOVERY DATA FOR SEMIVOLATILE COMPOUNDS SPIKED INTO TRIPLICATE SAMPLES OF AQUEOUS WASTE NO. 303 FROM ROSS SITE

Compounds	Found in[a] unspiked sample µg/g	Amount added µg/g	Amount found in spiked samples[a] µg/g	RSD of amount found in spiked samples (%)	% Recovery
N,N-dimethylacetamide	220	46	380	26	350
Phenol	140	280	390	24	89
2,4-Dimethylphenol	0.8	1.6	3.4	87	160
Naphthalene	0.9	1.8	2.3	5	78
Dimethylphthalate	ND	990	760	17	77
1,2,4-Trichlorobenzene	ND	500	400	25	80
Decafluorobiphenyl	-	500	390	25	78
D_8-Naphthalene	-	500	760	4	150
D_{12}-Chrysene	-	500	69	67	14

[a] Average of three replicates.

TABLE L-16. RECOVERY DATA FOR SEMIVOLATILE COMPOUNDS SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 304 FROM ROSS SITE

Compounds	Found in unspiked sample, μg/g[a]	Amount added μg/g	Amount found in spiked samples[a] μg/g	RSD of amount found in spiked samples (%)	% Recovery
N,N-dimethylacetamide	ND	500	890	11	180
Phenol	ND	2,700	8,200	3	300
2,4-Dimethylphenol	2,000	4,000	7,400	6	140
Naphthalene	490	990	2,500	3	200
Dimethylphthalate	ND	500	700	15	140
1,2,4-Trichlorobenzene	ND	500	660	3	130
Decafluorobiphenyl	-	500	530	3	110
D_8-Naphthalene	-	500	760	4	150
D_{12}-Chrysene	-	500	69	67	14

[a] Average of three replicates.

TABLE L-17. RECOVERIES OF SEMIVOLATILE ANALYTES FROM DUPLICATE SPIKED WATER AND DUPLICATE SPIKED XAD RESIN. SPIKING LEVEL: 20 µg/ℓ or 20 µg/75 g XAD, RESPECTIVELY

Compound	Spiked water			Spiked XAD		
	Spiking level µg/ℓ	Average recovery, %	% RSD	Spiking level µg/75 g XAD	Average recovery, %	% RSD
N,N-dimethylacetamide	100[a]	3.8[a]	35[a]	20	65	22
Phenol	20	42	40	20	106	59
Isophorone	20	101	26	20	86	43
Decafluorobiphenyl	20	77	31	20	80	49
2,4-Dimethylphenol	20	93	28	20	97	55
1,2,4-Trichlorobenzene	20	88	30	20	80	46
D_8-Naphthalene	20	88	23	20	79	39
Naphthalene	20	94	22	20	97	43
Dimethylphthalate	20	110	28	20	99	47
Diethylphthalate	20	112	28	20	101	42
Hexachlorobenzene	20	106	29	20	91	48
D_{12}-Chrysene	20	86	17	20	40	44
Bis(2-ethylhexylphthalate	20	49	26	20	88	58

[a] Obtained from separate recovery experiment.

TABLE L-18. RECOVERIES OF SEMIVOLATILE SURROGATE COMPOUNDS IN ROSS SAMPLES (% RECOVERY)

Sample type and number	Decafluorobiphenyl	D_8-Naphthalene	D_{12}-Chrysene
Waste, aqueous			
103	60	56	56
203	76	72	68
303-2B1	76	72	68
303-3B1	34	22	92
303-3B2	48	21	96
Waste, organic			
104	68	60	77
204	72	64	77
304-2B2	76	64	72
304-2B3	68	88	72
304-2B1	72	64	72
Waste to kiln, composited			
510	100	110	80
511	80	110	84
512	110	100	88
Waste to chamber, composited			
513	140	160	120
514	130	140	120
515	84	110	88
Blank XAD			
335	48	60	43
Front XAD + condensate			
507	60	65	Not added
508	50	65	Not added
509	60	50	Not added
Back XAD			
117	47	55	Interference
217	43	55	Interference
317	19	25	40
Filter blank			
234	Not added	Not added	90
Probe rinse and filter			
516	Not added	Not added	85
517	Not added	Not added	120
518	Not added	Not added	110

(continued)

TABLE L-18. (continued)

Sample type and number	Decafluorobiphenyl	D_8-Naphthalene	D_{12}-Chrysene
Water samples			
108	Not added	Not added	95
208	Not added	Not added	100
308	Not added	Not added	110
110	20	46	65
210	50	60	95
310	28	46	75
209	60	95	50
211	50	80	65
212	50	80	80
Chamber ash			
206	90	120	43
Spiked water			
534	60	73	75
535	94	102	96
Spiked XAD			
532	108	101	27
533	52	57	52
Spiked aqueous waste			
Average of 3 values	78	150	14
Spiked organic waste			
Average of 3 values	110	150	14

TABLE L-19. RESULTS OF ANALYSIS FOR METALS IN DUPLICATE SAMPLES OF AQUEOUS WASTE NO. 303 FROM ROSS SITE (μg/g)

Element	Rep 1	Rep 2	Average	% RSD
B	7.6	11	9.3	26
Ba	1.0	.98	.99	1.4
Ca	54	55	55	1.3
Cr	< .07	.36	NA	NA
Cu	.93	1.2	1.1	18
Fe	13	18	16	23
Fe	21	22	22	3.3
Mg	7.3	7.5	7.4	1.0
Mn	.89	.92	.91	2.3
Na	1,200	1,200	1,200	0
P	11	11	11	0
Pb	< 1.4	1.5	NA	NA
Sn	1.1	< 1.1	NA	NA
Zn	.75	.78	.77	2.8

TABLE L-20. RESULTS OF ANALYSIS FOR METALS IN DUPLICATE SAMPLES OF ORGANIC WASTE NO. 304 FROM ROSS SITE (μg/g)

Element	Rep 1	Rep 2	Average	% RSD
B	7.7	12	9.9	31
Ba	1,200	1,200	1,200	0
Ca	220	220	220	0
Cd	15	14	15	4.8
Co	3.6	3.2	3.4	8.3
Cr	420	420	420	0
Cu	80	80	80	0
Fe	280	240	260	11
Fe	290	240	270	13
K	13	8.4	11	30
Mg	73	76	75	2.8
Mn	8.6	8.5	8.6	8.2
Mo	51	37	44	22
Na	1,800	1,800	1,800	0
Ni	27	27	27	0
P	130	130	130	0
Pb	1,800	1,700	1,800	4.0
Sb	370	140	260	64
Sn	40	27	34	27
Tl	60	28	44	51
Zn	220	200	210	6.7

TABLE L-21. RECOVERY DATA FOR METALS SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 304 FROM ROSS SITE (μg/g)

		Rep 1			Rep 2			Rep 3		
Element	Unspiked	Spiked	Spike Level	% Recovery	Spiked	Spike level	% Recovery	Spiked	Spike level	% Recovery
Ag	< 0.3	< 0.3	55	a	< 0.3	66	a	< 0.3	65	a
As	< 14.0	74.6	58	110	88	70	110	100	68	130
Ba	1,200	1,200	57	b	1,300	69	b	1,600	68	b
Be	< 0.2	47	54	87	56	66	86	65	64	100
Cd	15	57	51	81	64	62	79	78	60	100
Cr	430	470	57	b	480	68	b	630	67	b
Hg	< 4.0	56	57	91	66	69	92	76	67	110
Ni	27	77	56	90	93	67	99	100	66	120
Pb	1,800	1,700	56	b	1,640	67	b	2,260	66	b
Sb	369	319	53	b	390	64	b	430	62	b
Se	< 21	83	53	130[c]	93	64	120[c]	110	62	150[c]
Tl	< 9.0	61	57	93	74	69	96	82	68	107

a Ag precipitated as AgCl during sample preparation.

b Spike level too low to be detected.

c Accuracy affected by large Ca interferences on Se emission peak.

TABLE L-22. RECOVERY DATA FOR METALS SPIKED INTO AQUEOUS WASTE NO. 303 FROM ROSS SITE (μg/g)

Metal	Unspiked	Spiked	Spike level	% Recovery
Ag	< 0.2	< 0.2	8.3	[a]
As	< 15.0	< 15.0	7.6	[a]
Ba	1.2	14	8.7	150
Be	< 0.3	12	8.2	140
Cd	< 0.03	11	7.7	150
Cr	< 0.03	13	8.6	150
Hg	< 3.00	13	8.6	150
Ni	< 0.063	12	8.3	150
Pb	2.3	15	8.4	150
Sb	< 0.18	11	8.0	140
Se	< 2.0	15	8.0	180
Tl	< 6.00	13	8.6	150

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[a] Spike level too low to be detected.

TABLE L-23. RECOVERY DATA FOR METALS SPIKED INTO SCRUBBER EFFLUENT NO. 310 FROM ROSS SITE (μg/g)

Metal	Unspiked	Spiked	Spike level	% Recovery
Ag	< 200	370	5,200	b
As	< 15,000	< 15,000	4,900	a
Ba	1,500	5,800	5,500	80
Be	< 300	4,300	5,200	82
Cd	1,200	5,300	4,900	85
Cr	740	5,000	5,400	79
Hg	< 3,000	5,000	5,900	88
Ni	440	4,600	5,300	78
Pb	12,000	16,000	5,300	70
Sb	3,800	7,800	5,000	80
Se	< 2,000	6,100	5,000	93
Tl	< 6,000	< 6,000	5,400	a

a Spike level too low to be detected.

b Ag precipitated as AgCl during sample preparation.

TABLE L-24. RECOVERY DATA FOR METALS SPIKED INTO DRUM WASTE NO. 527 FROM ROSS SITE (μg/g)

Metal	Unspiked	Spiked	Spike level	% Recovery
Ag	< 0.03	< 0.03	3.3	b
As	< 3.5	< 3.5	3.0	a
Ba	1.6	4.1	3.4	72
Be	< 0.15	1.5	3.2	48
Cd	< 0.016	1.3	3.1	44
Cr	2.4	5.9	3.4	100
Hg	< 3.0	< 3.0	3.4	a
Ni	8.8	15	3.3	175
Pb	0.31	1.4	3.3	34
Sb	< 3.00	< 3.0	3.2	a
Se	< 20.0	< 20.0	3.2	a
Tl	< 4.00	5.5	3.4	a

a Spike level too low to be detected.

b Ag precipitated as AgCl during sample preparation.

TABLE L-25. RESULTS OF ANALYSIS OF TRIPLICATE EXTRACTS OF ORGANIC WASTE NO. 301 FROM PLANT B SITE FOR VOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Chloroform	310	250	250	270	13
Carbon tetrachloride	2,000	1,600	2,000	1,900	12
Trichloroethylene	2,200	2,300	2,200	2,200	3
Benzene	200	140	110	150	31
Tetrachloroethylene	9,900	9,900	9,600	9,800	2
Toluene	44,000	46,000	46,000	45,000	3

TABLE L-26. RESULTS OF ANALYSIS OF TRIPLICATE EXTRACTS OF AQUEOUS WASTE NO. 302 FROM PLANT B SITE FOR VOLATILE POHCS (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Chloroform	21	22	23	22	5
Trichloroethylene	83	84	87	85	2
Benzene	7	7	7	7	0
Tetrachloroethylene	1,200	1,200	1,200	1,200	0
Toluene	2,500	2,600	2,900	2,700	8

TABLE L-27. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 301 FROM PLANT B SITE

Compound	Found in[a] unspiked sample μg/g	Amount added μg/g	% Recovery				
			Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Carbon tetrachloride	1,900	4,000	73	53	25	50	48
Trichloroethylene	2,200	4,400	100	68	95	88	20
Chloroform	270	620	63	66	40	56	25
Benzene	150	400	88	68	55	70	24
Tetrachloroethylene	9,800	19,800	87	75	59	74	19
D_4-1,2-Dichloroethane	-	250	100	130	72	100	29
D_{10}-Ethylbenzene	-	250	95	110	86	97	12

[a] Average of three replicates.

TABLE L-28. RECOVERIES OF VOLATILE SURROGATE COMPOUNDS IN PLANT B SAMPLES (% RECOVERY)

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Scrubber effluents	105	90	70
	205	100	70
	305	85	65
	405	95	70
	505	90	95
Organic waste	101	230	110
	201	150	120
	301-1	200	110
	301-2	170	110
	301-3	220	87
	401	190	93
	501	130	94
Aqueous waste	102	90	88
	202	110	100
	302-1	98	100
	302-2	100	110
	302-3	110	110
	402	100	110
	502	110	97
Ash	103	110	97
	203	100	97
	303	120	120
	403	120	70
	503	120	73

TABLE L-29. RESULTS OF ANALYSES OF REPLICATE EXTRACTS OF ORGANIC WASTE SAMPLE NO. 301 FROM PLANT B SITE FOR SEMIVOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Phenol[a]	1,400	1,800	1,900	1,700	14
Decafluorobiphenyl[a]	320	320	390	340	11
1,2,4-Trichlorobenzene[a]	81	77	83	80	4
D_8-Naphthalene[a]	330	330	410	360	13
Naphthalene[a]	430	430	480	450	6
Dimethylphthalate[a]	130	120	110	120	8
Dodecanol[a]	730	680	560	660	13
Diethylphthalate[a]	1,400	1,300	1,200	1,300	8
Hexachlorobenzene[a]	31	28	41	33	20
Butylbenzylphthalate[a]	490	490	360	450	17
D_{12}-Chrysene[a]	54	68	59	60	12
Bis(2-ethylhexylphthalate[a]	95	91	81	89	8
Ethylhexylchloride[b]	43,000	43,000	42,000	43,000	1
Isophorone[b]	230	210	250	230	9
Caprolactam[b]	30	ND	ND	NA	NA
C_4-alkylphenol[b]	72	69	64	68	6
Diphenylether[b]	2,000	2,800	2,300	2,400	17
Dimethylterephthalate[b]	150	150	150	150	0
Octachlorocyclopentene[b]	93	78	75	82	12
Isodrin[b]	64	59	35	53	29

a Quantified by comparison with an authentic standard measured same day.

b Quantified by using an estimated response factor.

NA = Not applicable.

TABLE L-30. RESULTS OF ANALYSES OF REPLICATE EXTRACTS OF AQUEOUS WASTE SAMPLE NO. 302 FROM PLANT B SITE FOR SEMIVOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Phenol[a]	3,300	2,200	3,200	2,900	21
Decafluorobiphenyl[a]	290	240	360	300	20
1,2,4-Trichlorobenzene[a]	45	36	55	45	21
D_8-Naphthalene[a]	340	280	420	350	20
Naphthalene[a]	47	41	59	49	19
Dimethylphthalate[a]	57	63	67	62	8
Dodecanol[a]	22	23	24	23	4
Diethylphthalate[a]	240	230	250	240	5
Butylbenzylphthalate[a]	ND	25	ND	8	173
D_{12}-Chrysene[a]	120	210	87	140	46
Bis(2-ethylhexylphthalate[a]	ND	26	ND	9	173
Ethylhexylchloride[b]	4,400	3,300	6,500	4,700	34
Isophorone[b]	88	9.2	ND	NA	NA
Caprolactam[b]	1,300	1,700	2,000	1,700	21
C_4-alkylphenol[b]	62	85	ND	NA	NA
Diphenylether[b]	820	1,100	11	640	88
Dimethylterephthalate[b]	5.8	8.3	8.4	7.5	20
Isodrin[b]	2.7	ND	ND	NA	NA

a Quantified by comparison with an authentic standard measured same day.

b Quantified by using an estimated response factor.

NA = Not applicable.

TABLE L-31. RECOVERY DATA FOR SEMIVOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 301 FROM PLANT B SITE

Compound	Measured in unspiked sample[a] (μg/g)	Added (μg/g)	% Recovery				% RSD
			Spike No. 1	Spike No. 2	Spike No. 3	Average	
Phenol	1,700	680	160	220	150	180	21
Decafluorobiphenyl	-	250	96	140	110	120	19
1,2,4-Trichlorobenzene	80	32	0	130	94	75	90
D_8-Naphthalene	-	250	80	140	120	110	27
Naphthalene	450	180	0	140	88	76	73
Hexachlorobenzene	33	13	0	100	140	80	90
D_{12}-Chrysene	-	250	72	32	35	46	48

[a] Average of three replicates.

TABLE L-32. RECOVERY DATA FOR SEMIVOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF AQUEOUS WASTE NO. 302 FROM PLANT B SITE

Compound	Measured in unspiked sample[a] (μg/g)	Added (μg/g)	% Recovery				% RSD
			Spike No.1	Spike No. 2	Spike No. 3	Average	
Phenol	2,900	1,010	170	250	280	230	24
Decafluorobiphenyl	-	250	88	100	120	100	16
1,2,4-Trichlorobenzene	45	18	0	33	39	24	87
D_8-Naphthalene	-	250	76	92	100	89	14
Naphthalene	49	20	0	0	0	0	0
Hexachlorobenzene	ND	100	80	96	100	92	12
D_{12}-Chrysene	-	250	110	120	76	100	23

[a] Average of three replicates.

TABLE L-33. RECOVERIES OF SELECTED SEMIVOLATILE ANALYTES FROM SPIKED REAGENT WATER. SPIKING LEVEL = 20 μg/ℓ

Compound	% Recovery			Average	% RSD
	Spike No. 1	Spike No. 2	Spike No. 3		
Phenol	29	23	22	25	15
Decafluorobiphenyl	20	15	14	16	20
1,2,4-Trichlorobenzene	38	30	27	32	18
D_8-Naphthalene	46	35	33	38	18
Naphthalene	49	36	35	40	20
Dimethylphthalate	90	47	60	66	34
Dodecanol	92	52	69	71	28
Diethylphthalate	98	65	80	81	20
Hexachlorobenzene	96	73	82	84	14
Butylbenzylphthalate	130	150	180	150	16
D_{12}-Chrysene	109	130	139	126	12
Bis(2-ethylhexylphthalate	129	170	182	160	17

TABLE L-34. RECOVERIES OF SELECTED SEMIVOLATILE ANALYTES FROM TRIPLICATE SPIKED XAD RESIN. SPIKING LEVEL = 20 μg/75 g XAD

Compound	% Recovery				
	Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Phenol	75	78	52	68	21
Decafluorobiphenyl	78	84	46	69	29
1,2,4-Trichlorobenzene	59	62	36	52	27
D_8-Naphthalene	59	64	38	54	26
Naphthalene	101	107	68	92	23
Dimethylphthalate	102	94	97	98	4
Dodecanol	98	92	97	96	3
Diethylphthalate	103	98	101	101	2
Hexachlorobenzene	98	100	100	99	1
Butylbenzylphthalate	141	134	120	132	8
D_{12}-Chrysene	73	60	56	63	14
Bis(2-ethylhexyl)phthalate	100	86	79	88	12

TABLE L-35. RECOVERIES OF SURROGATE COMPOUNDS IN SAMPLES ANALYZED FOR SEMIVOLATILE POHCs AND PICs FROM PLANT B SITE (% RECOVERY)

Sample No.	Sample type	Decafluorobiphenyl	D_8-Naphthalene	D_{12}-Chrysene
101	Organic waste	140	150	44
201	Organic waste	160	170	13
301	Organic waste	130	130	22
301	Organic waste	130	130	27
301	Organic waste	150	160	24
401	Organic waste	100	110	36
501	Organic waste	120	130	28
102	Aqueous waste	100	120	76
202	Aqueous waste	110	130	37
302	Aqueous waste	120	140	48
302	Aqueous waste	96	110	84
302	Aqueous waste	140	170	35
402	Aqueous waste	92	100	64
502	Aqueous waste	110	130	52
Water samples				
514	Water blank	70	95	195
405	Scrubber effluent	60	90	55
505	Scrubber effluent	105	135	35
Effluent samples				
513	Glassware/solvent blank	80	90	50
274	XAD blank	60	65	26
109	Front XAD	48	Not added	Not added
209	Front XAD	20	Not added	Not added
309	Front XAD	70	Not added	Not added
110	Back XAD	55	60	50
210	Back XAD	50	55	95
310	Back XAD	170	95	16

(continued)

TABLE L-35. (continued)

Sample No.	Sample type	Decafluorobiphenyl	D_8-Naphthalene	D_{12}-Chrysene
108	Condensate	37	47	135
208	Condensate	41	60	95
308	Condensate	55	70	75
510	Front XAD and condensate - Run 1	70	70	Not added
511	Front XAD and condensate - Run 2	215	395	Not added
512	Front XAD and condensate - Run 3	100	95	Not added
133	Back XAD	85	85	50
233	Back XAD	85	80	46
333	Back XAD	65	65	50
103	Ash	96	80	80
203	Ash	100	80	96
303	Ash	110	92	80
403	Ash	100	84	76
503	Ash	100	84	72
501	Probe rinse and filter	Not added	Not added	90
502	Probe rinse and filter	Not added	Not added	32
503	Probe rinse and filter	Not added	Not added	32
507	Probe rinse and filter	Not added	Not added	75
508	Probe rinse and filter	Not added	Not added	70
509	Probe rinse and filter	Not added	Not added	43
273	Filter blank	Not added	Not added	80
504-S1	Spiked water	20	46	109
504-S2	Spiked water	15	35	130
504-S3	Spiked water	14	33	139
503-S1	Spiked XAD	78	59	73
503-S2	Spiked XAD	84	64	60
503-S3	Spiked XAD	46	38	56

(continued)

TABLE L-35. (continued)

Sample No.	Sample type	Decafluorobiphenyl	D_8-Naphthalene	D_{12}-Chrysene
302-S1	Spiked aqueous waste	88	76	110
302-S2	Spiked aqueous waste	100	92	120
302-S3	Spiked aqueous waste	120	100	76
301-S1	Spiked organic waste	96	80	72
301-S2	Spiked organic waste	140	140	32
301-S3	Spiked organic waste	110	120	35

TABLE L-36. RESULTS OF REPLICATE ANALYSES FOR METALS IN REAGENT BLANKS AND ORGANIC WASTE NO. 301 FROM PLANT B SITE (μg/g)

Element	Reagent Blank			301-1C					301-1C-4			
	Rep 1	Rep 2	Average	Rep 1	Rep 2	Rep 3	Average	% RSD	Rep 1	Rep 2	Average	% RSD
Al	< 5.2	< 5.2	< 5.2	510	530	480	510	5.0	630	280	460	54
Al	< 4.8	< 4.8	< 4.8	510	530	480	510	5.0	630	280	460	54
B	< 2.9	< 0.35	NA	24	21	25	23	8.9	< 0.75	< 0.75	< 0.75	NA
Ba	< 2.0	< 2.0	< 2.0	140	140	140	140	0	170	91	130	42
Ca	< 20	< 20	< 20	> 6,100	> 6,200	> 5,900	> 6,100	NA	13,000	12,000	13,000	5.6
Cd	< 1.0	< 1.0	< 1.0	6.1	5.6	5.2	5.6	8.0	7.0	4.6	5.8	29
Co	< 1.0	< 1.0	< 1.0	3.6	3.3	3.2	3.4	6.2	2.0	< 1.3	NA	NA
Cr	< 1.0	< 1.0	< 1.0	59	58	54	57	4.6	70	40	55	39
Cu	< 1.0	< 1.0	< 1.0	44	43	42	43	2.3	48	37	43	18
Fe	< 9.5	< 9.5	< 9.5	1,300	1,400	1,500	1,400	7.1	1,800	900	1,400	47
Fe	< 13	< 13	< 13	850	1,100	830	930	16	1,800	930	1,400	45
Hg	< 10	< 10	< 10	< 10	< 10	< 10	< 10	NA	10	7.5	9.0	20
K	< 10	< 10	< 10	98	53	75	75	30	< 250	< 250	< 250	NA
Mg	< 1.4	< 1.4	< 1.4	190	190	180	190	3.1	210	190	200	7.1
Mn	< 1.0	< 1.0	< 1.0	13	12	13	13	4.6	14	9.5	12	27
Mo	< 1.0	< 1.0	< 1.0	8.2	8.1	8.0	8.1	1.2	5.3	2.6	4.0	48
Na	< 12	< 12	< 12	940	880	940	920	3.8	860	1,200	1,000	23
Ni	< 1.0	< 1.0	< 1.0	7.5	7.1	7.0	7.2	3.7	5.1	3.2	4.2	32
P	< 37	< 37	< 37	> 5,700	> 5,500	> 5,200	> 5,500	NA	6,200	4,000	5,100	30
Pb	< 10	< 10	< 10	160	150	140	150	6.7	190	93	140	48
Sb	< 10	< 10	< 10	64	60	58	61	5.0	78	56	67	23
Se	< 100	< 100	< 100	< 100	160	< 100	NA	NA	1,100	1,000	1,100	6.7
Sn	< 1.0	< 1.0	< 1.0	41	42	39	41	3.8	47	23	35	48
Ti	< 1.2	< 1.2	< 1.2	58	32	39	43	31	20	73	47	81
Zn	< 1.4	< 1.4	< 1.4	190	170	160	170	8.8	230	140	190	34

TABLE L-37. RESULTS OF REPLICATE ANALYSES FOR METALS IN REAGENT BLANKS AND AQUEOUS WASTE NO. 302 FROM PLANT B SITE (μg/g)

Element	Aqueous reagent blank							
	Rep 1	Rep 2	Average	Rep 1	Rep 2	Rep 3	Average	% RSD
Al	< 5.2	< 5.2	< 5.2	46	46	46	46	0
Al	< 4.8	< 4.8	< 4.8	47	47	47	47	0
B	5.6	3.7	4.7	150	150	150	150	0
Ba	< 2.0	< 2.0	< 2.0	6.6	6.7	6.7	6.7	8.6
Ca	< 20	< 20	< 20	360	370	360	360	1.6
Cr	< 1.0	< 1.0	< 1.0	2.9	2.9	2.9	2.9	0
Cu	< 1.0	< 1.0	< 1.0	1,200	1,200	1,200	1,200	0
Fe	< 9.5	< 9.5	< 9.5	140	150	140	140	4.0
Fe	< 13	< 13	< 13	150	160	160	160	3.7
K	< 10	< 10	< 10	300	300	290	300	1.9
Mg	< 1.4	< 1.4	< 1.4	77	77	77	77	0
Mn	< 1.0	< 1.0	< 1.0	2.2	2.3	2.3	2.3	2.5
Na	< 12	< 12	< 12	1,900	1,900	1,900	1,900	0
Ni	< 1.0	< 1.0	< 1.0	2.4	2.3	2.3	2.3	2.5
P	< 37	< 37	< 37	1,300	1,300	1,200	1,300	4.6
Pb	< 10	< 10	< 10	11	12	11	11	5.1
Sn	< 1.0	< 1.0	< 1.0	11	12	12	12	4.9
Ti	< 1.2	< 1.2	< 1.2	1.4	1.5	1.4	1.4	4.0
Zn	< 1.4	< 1.4	< 1.4	97	97	94	96	1.8

TABLE L-38. RECOVERY DATA FOR METALS SPIKED INTO ORGANIC WASTE NO. 301 FROM PLANT B SITE (μg/g)

Element	Unspiked	Spike level	Spike No. 1	% Recovery	Spike No. 2	% Recovery	Average % recovery	Unspiked No. 301-1 repeat	Spike level	Repeat spike No. 1	% Recovery	Repeat spike No. 2	% Recovery	Average % recovery
As	< 20	43	51	120	73	170	150	< 2.3	-	< 2.3	-	< 2.3	-	-
Ba	140	43	180	93	200	140	120	170	500	560	78	490	64	71
Be	< 1.0	40	39	98	60	150	120	< 0.50	-	< 0.50	-	< 0.50	-	-
Cd	6.1	38	39	86	58	140	110	7.0	-	3.8	-	3.5	-	-
Cr	59	42	92	79	110	120	100	70	200	220	75	190	60	68
Hg	< 10	42	42	100	63	150	130	10	-	< 7.5	-	< 7.5	-	-
Ni	7.5	42	43	84	63	130	110	5.1	-	< 2.3	-	< 2.3	-	-
Pb	160	41	180	49	210	120	85	190	490	530	69	470	57	63
Sb	64	39	94	77	110	120	97	78	180	240	90	220	79	85
Se	< 100	39	260	670	120	300	480	1,100	-	1,200	-	1,000	-	-
Tl	< 20	42	48	110	68	160	140	< 13	-	< 13	-	< 13	-	-

TABLE L-39. RECOVERY DATA FOR METALS SPIKED INTO AQUEOUS WASTE NO. 302 FROM PLANT B SITE

Element	Unspiked	Spike level	Spike No. 1	% Recovery	Spike No. 2	% Recovery	Average % recovery
Ag	< 1.0	85	1.2	a	< 1.0	a	a
As	< 20	90	90	100	73	81	91
Ba	6.6	89	98	100	78	80	90
B	< 1.0	85	75	88	62	73	81
Cd	< 1.0	80	69	86	57	71	79
Cr	2.9	88	88	97	71	77	87
Hg	< 10	89	81	91	67	75	83
Ni	2.4	87	83	93	67	74	84
Pb	11	86	83	84	71	70	77
Sb	< 10	82	76	93	63	77	85
Se	< 100	82	110	130	< 100	NA	NA
Tl	< 20	89	86	97	68	76	87

a Ag lost due to AgCl precipitation during sample preparation.

NA = Not applicable.

TABLE L-40. RECOVERY DATA FOR METALS SPIKED INTO ASH SAMPLE NO. 203 FROM PLANT B SITE (μg/g)

Element	Unspiked	Spike level	Spike No. 1	% Recovery	Spike No. 2	% Recovery	Average % recovery
Be	< 7.0	9.1	8.7	86	11	110	98

TABLE L-41. RESULTS OF ANALYSIS OF REPLICATE EXTRACTS OF LIQUID WASTE NO. 301 FROM UPJOHN SITE FOR VOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Chloroform	37	42	42	40	7
Carbon tetrachloride	17,000	20,000	21,000	19,000	11
Trichloroethylene	14,000	16,000	16,000	15,000	7.5
Benzene	25	24	20	23	12
Toluene	130	130	80	110	25

TABLE L-42. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF LIQUID WASTE NO. 301 FROM UPJOHN SITE

Compound	Found in[a] unspiked sample µg/g	Amount added µg/g	% Recovery Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Carbon tetrachloride	19,000	19,000	410	470	440	440	6.8
Trichloroethylene	15,300	15,300	370	440	330	380	15
Toluene	110	220	540	400	850	600	39
Chloroform	40	80	2,290	1,700	2,800	2,260	24
D_4-1,2-Dichloroethane	-	250	ND	ND	ND	ND	NA
D_{10}-Ethylbenzene	-	250	98	70	96	88	18

[a] Average of three determinations.

ND = Not detected due to small amount of sample analyzed.

NA = Not applicable.

TABLE L-43. RECOVERIES OF VOLATILE SURROGATE COMPOUNDS IN UPJOHN SAMPLES (% RECOVERY)

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Make-up waters	205	150	130
	305	130	120
	405	100	110
Waste feed	201	110	25
	301-1	83	37
	301-2	100	32
	301-3	81	32
	401	88	29

TABLE L-44. RESULTS OF ANALYSIS OF TRIPLICATE EXTRACTS OF WASTE SAMPLE NO. 301 FROM UPJOHN SITE FOR SEMIVOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
m-Dichlorobenzene[a]	2,400	1,900	2,600	2,300	16
p-Dichlorobenzene[a]	59,000	50,000	69,000	59,000	16
o-Dichlorobenzene[a]	47,000	41,000	49,000	46,000	9
Decafluorobiphenyl[a]	130	120	140	130	8
1,2,4-Trichlorobenzene[a]	300	240	320	290	15
D_8-Naphthalene[a]	170	140	170	160	11
D_{12}-Chrysene[a]	46	43	24	38	32
Bis(2-ethylhexyl)phthalate	560	520	420	500	14
Phenyl isocyanate[b]	ND[c]	160,000	160,000	160,000[e]	0[e]
Aniline[b]	13,000	ND[d]	14,000	14,000[e]	5[e]

[a] Quantified by comparison with authentic standard run same day, utilizing internal standard.

[b] Quantified by utilizing an estimated GC/MS response factor.

[c] Compound missed by late venting of solvent during GC/MS analysis.

[d] Not determinable. Possibly obscured by other compounds. Possibly not present.

[e] For two values only.

TABLE L-45. RESULTS OF REPEATED ANALYSES OF SAMPLE 501 (FRONT XAD PLUS CONDENSATE)EXTRACT FROM UPJOHN INCINERATOR SITE (μg)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Phenol[a]	21	21	24	22	8
m-Dichlorobenzene[a]	220	210	200	210	4.8
p-Dichlorobenzene[a]	74	86	66	75	14
o-Dichlorobenzene[a]	36	36	35	35	1.6
Decafluorobiphenyl[a]	8.9	8.7	12	9.9	19
1,2,4-Trichlorobenzene[a]	98	99	120	106	12
D_8-Naphthalene[a]	11	10	12	11	9
Naphthalene[a]	40	40	7.9	29	63
Hexachlorobenzene[a]	1.3	1.3	1.8	1.5	20
Bis(2-ethylhexyl)phthalate[a]	18	19	15	17	12
o-Chlorophenol[b]	13	13	9.2	12	19
Trichlorophenol[b]	280	280	280	280	0
Pentachlorophenol[b]	26	30	25	27	9
o-Nitrophenol[b]	ND[c]	ND[c]	30	10	NA

[a] Quantified by comparison with authentic standard run same day and utilizing internal standard.

[b] Quantified by comparison with authentic standard run recently to obtain measured response factor.

[c] Not determinable. Possibly obscured by impurities. Possibly not present.

NA = Not applicable.

TABLE L-46. RECOVERY DATA FOR SEMIVOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF LIQUID WASTE NO. 301 FROM UPJOHN SITE

Compound	Found in unspiked sample[a] μg/g	Amount added μg/g	% Recovery Spike No. 1	% Recovery Spike No. 2	% Recovery Spike No. 3	Average	% RSD
m-Dichlorobenzene	2,300	4,800	67	83	0	50	88
1,2,4-Trichlorobenzene	290	480	110	130	110	120	9.9
Naphthalene	160	500	74	92	78	81	12
2,6-Toluene diisocyanate	ND	500	80	90	84	85	5.9
Bis(2-ethylhexyl)phthalate	500	1,100	0	0	0	0	0
Decafluorobiphenyl	-	250	48	52	48	49	4.7
D_8-Naphthalene	-	250	84	100	88	91	9.2
D_{12}-Chrysene	-	250	0	0	0	0	0

[a] Average of three replicates.

TABLE L-47. RECOVERIES OF SELECTED SEMIVOLATILE ANALYTES FROM TRIPLICATE SPIKED WATER (FOR UPJOHN SITE)

Sample No.: Compound	μg Added	Spike No. 1, No. 510		Spike No. 2, No. 511		Spike No. 3, No. 512		Average recovery	% RSD
		μg found	% R	μg found	% R	μg found	% R		
Phenol	50	17	34	18	36	18	36	35	3.3
m-Dichlorobenzene	20	15	75	10	50	16	80	68	24
o-Dichlorobenzene	20	20	100	15	75	22	110	95	19
p-Dichlorobenzene	20	16	80	12	60	17	85	75	18
1,2,4-Trichlorobenzene	20	15	75	14	70	17	85	77	10
Naphthalene	20	18	90	16	80	19	95	88	8.6
Decafluorobiphenyl	20	11	55	10	50	12	60	55	9.1
D_8-Naphthalene	20	21	110	20	100	23	120	110	9.1
D_{12}-Chrysene	20	19	95	20	100	15	75	90	15
Toluene 2,6-diisocyanate[a]	20	0	0	0	0	0	0	0	0

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[a] Determined in separate experiment.

% R = Percent recovery.

TABLE L-48. RECOVERIES OF SELECTED SEMIVOLATILE ANALYTES FROM TRIPLICATE SAMPLES OFSPIKED XAD (FOR UPJOHN SITE)

Sample No.: Compound	µg Added	Spike No. 1, No. 507		Spike No. 2, No. 508		Spike No. 3, No. 509		Average recovery	% RSD
		µg found	% R	µg found	% R	µg found	% R		
Phenol	50	68	140	93	190	140	280	200	35
m-Dichlorobenzene	20	24	120	12	60	15	75	85	37
o-Dichlorobenzene	20	31	160	21	110	23	120	130	20
p-Dichlorobenzene	20	24	120	16	80	19	95	98	21
1,2,4-Trichlorobenzene	20	25	130	20	100	25	130	120	14
Naphthalene	20	83	420	34	170	43	220	270	49
Decafluorobiphenyl	20	19	95	15	75	19	95	88	13
D_8-Naphthalene	20	33	170	26	130	33	170	160	15
D_{12}-Chrysene	20	9.0	45	7.4	37	21	110	64	63
Toluene 2,6-diisocyanate[a]	20	18	90	18	90	18	90	90	0

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[a] Determined in separate experiment.

% R = Percent recovery.

TABLE L-49. RECOVERIES OF SEMIVOLATILE SURROGATE COMPOUNDS FROM UPJOHN SAMPLES (% RECOVERY)

Sample type	Sample No.	Decafluoro-biphenyl	D_8-Naphthalene	D_{12}-Chrysene
Liquid waste	101	44	52	12
	201	52	56	13
	301-3B-1	52	68	18
	301-3B-2	48	56	17
	301-3B-3	56	68	10
	401	76	80	48
Solvent blank	505	60	55	95
XAD blank	138	60	95	75
Front XAD and condensate	501	60	60	Not added
	502	75	55	Not added
	503	65	70	Not added
Back XAD	211	27	80	26
	311	31	46	18
	411	24	37	22
Water blank	514	70	95	190
Make-up water	205	44	90	65
	305	50	90	36
	405	28	55	35
Duplicate GC/MS runs	501	45	55	Not added
	501	44	50	Not added
Spiked waste feed	301-S1	48	84	0
	301-S2	52	100	0
	301-S3	48	88	0
Spiked water	510	55	110	95
	511	50	100	100
	512	60	120	75
Spiked XAD	507	95	170	45
	508	75	130	37
	509	95	170	110

TABLE L-50. RESULTS OF TRIPLICATE ANALYSES OF LIQUID WASTE NO. 201 FROM ZAPATA SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Methylene chloride	150	240	130	170	34
Carbon tetrachloride	7,500	7,000	7,300	7,300	3.5
Trichloroethylene	7,100	7,000	7,100	7,100	0.8
Benzene	ND	< 20	ND	< 20	NA
Toluene	2,300	5,500	2,100	3,300	58
Chlorobenzene	7,700	6,200	8,800	7,600	17

TABLE L-51. RECOVERIES OF SELECTED VOLATILE ANALYTES SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 201 FROM ZAPATA SITE

Compound	Found in unspiked sample µg/g	Amount added µg/g	% Recovery				
			Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Carbon tetrachloride	7,300	14,600	78	88	75	80	8.5
Trichloroethylene	7,100	14,200	100	130	100	110	16
Chlorobenzene	7,600	15,200	91	99	93	94	4.4
Methylene chloride	170	340	100	76	65	80	22
D_4-1,2-Dichloroethane	-	250	47	41	27	38	27
D_{10}-Ethylbenzene	-	250	100	97	120	110	12

TABLE L-52. RECOVERIES OF VOLATILE SURROGATE COMPOUNDS FROM SAMPLES FROM ZAPATA SITE

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Waste feed	101	71	100
	201-1	53	120
	201-2	50	120
	201-3	80	130
	301	80	99
	401	110	120

TABLE L-53. RESULTS OF ANALYSIS OF TRIPLICATE EXTRACTS OF WASTE FEED NO. 301 FROM AMERICAN CYANAMID SITE FOR VOLATILE POHCs (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Methylene chloride	< 1	< 1	< 1	< 1	0
Chloroform	< 1	< 1	< 1	< 1	0
1,1,1-Trichloroethane	ND	< 1	ND	< 1	NA
Trichloroethylene	ND	< 1	ND	< 1	NA
Benzene	< 1	< 1	< 1	< 1	0
Tetrachloroethylene	< 1	< 1	< 1	< 1	0
Toluene	3.6	4.5	3.0	3.7	20
Chlorobenzene	< 1	< 1	ND	< 1	NA

NA = Not applicable.

TABLE L-54. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF WASTE NO. 301 FROM AMERICAN CYANAMID SITE

Compound	Found in unspiked sample µg/g	Amount added µg/g	% Recovery Spike No. 1	% Recovery Spike No. 2	% Recovery Spike No. 3	% Recovery Average	% RSD
Carbon tetrachloride	ND	1,000	93	79	88	87	8.2
Trichloroethylene	< 1	1,000	120	110	120	120	4.9
Chloroform	< 1	1,000	91	98	110	100	9.6
Toluene	3.7	1,000	100	99	100	100	< 1
D_4-1,2-Dichloroethane	-	250	95	97	92	95	2.7
D_{10}-Ethylbenzene	-	250	88	82	NA	85	5.0

NA = Recovery not available due to computer failure.

TABLE L-55. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF WASTE NO. 401 FROM AMERICAN CYANAMID SITE

Compound	Found in unspiked sample μg/g	Amount added μg/g	% Recovery Spike No. 1	% Recovery Spike No. 2	% Recovery Spike No. 3	Average	% RSD
Carbon tetrachloride	ND	1,000	43	54	77	58	30
Trichloroethylene	ND	1,000	62	67	85	71	17
Chloroform	< 1	1,000	72	98	97	89	17
Benzene	28	60	80	90	110	93	16
D_4-1,2-Dichloroethane	-	250	88	100	82	90	10
D_{10}-Ethylbenzene	-	250	25	33	55	38	41

TABLE L-56. RECOVERIES OF VOLATILE SURROGATES FROM AMERICAN CYANAMID SAMPLES

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Waste feed	101	130	80
	201	130	53
	301-1	88	I.T.
	301-2	93	I.T.
	301-3	100	I.T.
	401	110	I.T.
	501	110	I.T.

I.T. = Co-eluting interfering compound caused saturation and truncation of the D_{10}-ethylbenzene peak and prevented its measurement.

TABLE L-57. RECOVERIES OF SELECTED SEMIVOLATILE POHC ANALYTES FROM SPIKED XAD

Compound	μg Added	Rep 1		Rep 2		Rep 3		Average recovery	% RSD
		μg found	% R	μg found	% R	μg found	% R		
Aniline	50	11	22	ND	0	ND	0	6.7	170
Phenol	50	85	170	120	240	175	350	250	36
Nitrobenzene	20	26	130	21	110	27	140	130	12
2-Aminophenol	100	325	325	300	300	interference	-	310	5.7
Naphthalene	20	79	400	32	160	40	200	250	51
Decafluorobiphenyl	20	27	140	21	110	27	140	130	13
D_8-Naphthalene	20	27	140	22	110	28	140	130	13
D_{12}-Chrysene	20	7.2	36	5.9	30	17	85	50	60

% R = Percent recovery.

TABLE L-58. RECOVERIES OF SELECTED SEMIVOLATILE POHCs FROM SPIKED WATER

Compound	μg Added	Rep 1		Rep 2		Rep 3		Average recovery	% RSD
		μg found	% R	μg found	% R	μg found	% R		
Aniline	50	37	74	34	68	42	84	75	11
Phenol	50	21	42	22	44	23	46	44	4.5
Nitrobenzene	20	17	85	15	75	19	95	85	12
2-Aminophenol	100	194	194	170	170	210	210	190	11
Naphthalene	20	17	85	16	80	18	90	85	5.9
Decafluorobiphenyl	20	16	80	15	75	18	90	82	9.4
D_8-Naphthalene	20	18	90	17	85	19	95	90	5.6
d_{12}-Chrysene	20	16	80	16	80	12	60	73	16

% R = Percent recovery.

TABLE L-59. RECOVERIES OF SEMIVOLATILE SURROGATE COMPOUNDS FROM ALL AMERICAN CYANAMID SAMPLES (% RECOVERY)

Sample type	Sample No.	Decafluoro-biphenyl	D_8-Naphthalene	D_{12}-Chrysene
Waste samples	201	100	110	94
	301	94	110	90
	401	94	100	99
	501	94	110	77
Effluent Samples				
XAD + condensate	902	95	120	46
	903	110	150	0
	904	0	110	110
	905	95	100	110
Filter(s) + probe rinse(s)	907	NA	NA	110
	908	NA	NA	180
	909	NA	NA	60
	910	NA	NA	49
Blanks				
XAD	519	55	95	45
Filter + probe rinse	911	32	35	160
Spiked XAD	507	140	140	36
	508	110	110	30
	509	140	140	85
Spiked water	510	80	90	80
	511	75	85	80
	512	90	95	60

TABLE L-60. RESULTS OF REPLICATE ANALYSES FOR METALS IN REAGENT BLANKS AND LIQUID WASTE NO. 301 FROM AMERICAN CYANAMID SITE

	Concentration µg/g				Concentration µg/g			
	Reagent blank				Sample 301-1C			
Element	Rep 1	Rep 2	Average	% RSD	Rep 1	Rep 2	Average	% RSD
Al	5.2	4.2	4.7	15	14	16	15	9.4
Al	5.8	4.7	5.3	15	15	16	16	4.6
As	0.75	0.92	0.84	14	11	< 11	NA	NA
B	32	28	30	9.4	33	66	50	47
Ba	0.02	0.02	0.02	0	< 0.24	0.28	NA	NA
Ca	4.4	7.3	5.9	35	440	290	370	29
Cd	0.01	0.02	0.02	47	< 0.84	< 0.84	< 0.84	NA
Co	< 0.005	< 0.005	< 0.005	NA	1.1	1.4	1.3	17
Cr	< 0.007	< 0.007	< 0.007	NA	250	260	260	2.8
Cu	< 0.004	< 0.004	< 0.004	NA	38	42	40	7.1
Fe	< 0.14	0.38	NA	NA	1,300	1,400	1,400	5.2
Fe	1.3	1.3	1.3	0	> 1,100	> 1,200	NA	NA
Hg	< 0.03	0.97	NA	NA	4.6	4.6	4.6	0
Mg	0.20	0.16	0.18	16	23	12	18	44
Mn	< 0.001	0.01	NA	NA	34	37	36	6.0
Mo	0.08	0.18	0.13	54	4.2	4.4	4.3	3.3
Na	50	45	48	7.4	70	110	90	31
Ni	0.04	0.09	0.07	54	170	190	180	7.9
P	3.0	2.9	3.0	2.4	5.8	9.4	7.6	34
Pb	0.05	0.76	0.41	120	< 1.3	1.7	NA	NA
Sb	0.51	0.40	0.46	17	< 2.4	< 2.4	< 2.4	NA
Se	1.3	0.64	0.97	48	10	< 10	NA	NA
Sn	0.66	0.74	0.70	8.1	8.0	8.0	NA	NA
Ti	< 0.003	0.03	NA	NA	< 0.16	0.23	NA	NA
Tl	0.68	< 0.05	NA	NA	5.2	< 5.2	NA	NA
Zn	0.26	0.25			43	24		

NA = Not applicable.

TABLE L-61. RECOVERY DATA FOR METALS SPIKED INTO LIQUID WASTE NO. 301 FROM AMERICAN CYANAMID SITE (µg/g)

Element	Unspiked	Spike level	Spike No. 1	% Recovery	Spike No. 2	% Recovery	Average % recovery
Ag	< 0.45	57	2.7	4.8	4.5	7.8	6.3
B	33	60	76	72	72	65	69
Ba	< 0.24	60	9.9	17	9.3	16	16
Be	< 0.51	58	57	98	53	91	95
Cd	< 0.84	58	60	100	55	95	98
Cr	250	59	320	120	310	100	110
Cu	38	57	99	110	94	98	100
Fe	1,300	250	1,600	120	1,500	80	100
Fe	> 1,100	250	> 1,300	NA	> 1,200	NA	NA
Hg	4.6	60	65	100	56	86	93
Ni	170	58	240	120	230	100	110
Pb	< 1.3	58	55	95	51	88	92
Sb	< 2.4	58	54	93	20	34	44
Se	10	55	62	95	66	100	98
Tl	5.2	60	65	100	64	98	99
Zn	43	57	73	53	77	60	57

NA = Not applicable.

TABLE L-62. RESULTS OF ANALYSIS OF THREE REPLICATES OF AQUEOUS WASTE EXTRACT NO. 401 FROM MITCHELL SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Methylene chloride	8.9	9.6	27	15	68
Chloroform	ND	ND	< 1	ND	NA
1,1,1-Trichloroethane	1.9	1.5	2.3	1.9	21
Trichloroethylene	< 1	< 1	1.5	≅ 1	NA
Tetrachloroethylene	< 1	< 1	< 1	< 1	0
Toluene	97	100	96	98	2.0

ND = Not detected.

NA = Not applicable.

TABLE L-63. RESULTS OF ANALYSES OF THREE REPLICATES OF ORGANIC WASTE NO. 402 FROM MITCHELL SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Methylene chloride	59	64	51	58	11
Carbon tetrachloride	5,200	7,000	5,800	6,000	15
Trichloroethylene	2,200	2,500	2,200	2,300	7.5
Benzene	75	89	90	85	10
Tetrachloroethylene	42	58	50	50	16
Toluene	a	a	a	a	NA

[a] Quantification for toluene was not possible due to a co-eluting compound which saturated the detector and truncated the ion peak.

NA = Not applicable.

ND = Not detected.

TABLE L-64. RECOVERY DATA FOR SELECTED VOLATILE ANALYTES SPIKED INTO TRIPLICATE SAMPLES OF AQUEOUS WASTE NO. 401 FROM MITCHELL SITE

Compound	Found in[a] unspiked sample µg/g	Amount added µg/g	% Recovery				
			Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Carbon tetrachloride	ND	1,000	12	14	10	12	17
Trichloroethylene	1	1,000	22	25	23	23	6.5
Toluene	98	200	1	< 1	0	< 1	NA
Chloroform	ND	1,000	47	51	53	50	6.1
D_4-1,2-Dichloroethane	-	250	60	62	59	60	2.5
D_{10}-Ethylbenzene	-	250	42	44	48	45	6.8

[a] Average of three replicate.

NA = Not applicable.

TABLE L-65. RECOVERY DATA FOR SELECTED VOLATILE ANALYTES SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 402 FROM MITCHELL SITE

Compound	Found in[a] unspiked sample µg/g	Amount added µg/g	% Recovery				
			Spike No. 1	Spike No. 2	Spike No. 3	Average	% RSD
Carbon tetrachloride	6,000	12,000	130	3	3	45	160
Trichloroethylene	2,300	4,600	190	170	190	180	6.3
Benzene	85	170	110	88	110	100	12
Tetrachloroethylene	50	100	45	70	49	55	25
D_4-1,2-Dichloroethane	-	250	77	100	100	92	14
D_{10}-Ethylbenzene	-	250	92	84	88	88	4.5

[a] Average of three replicates.

TABLE L-66. RECOVERIES OF VOLATILE SURROGATE COMPOUNDS IN MITCHELL SAMPLES

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Organic waste	102	53	120
	202	93	87
	302	ND	14
	402-1	85	91
	402-2	110	97
	402-3	91	96
Aqueous waste	101	120	33
	201	160	49
	301	170	54
	401-1	170	54
	401-2	180	55
	401-3	170	53
Ash	501	110	33

ND = Not detected due to small amount of sample analyzed.

TABLE L-67. RECOVERY DATA FOR SELECTED SEMIVOLATILE POHCs FROM SPIKED WATER (FOR MITCHELL SAMPLES)

Compound	μg Added	Rep 1		Rep 2		Rep 3		Average recovery	% RSD
		μg found	% R	μg found	% R	μg found	% R		
Phenol	50	22	44	23	46	24	48	46	4.3
2,4-Dimethylphenol	20	20	100	22	110	22	110	110	5.4
Naphthalene	20	17	85	16	80	18	90	85	5.9
Diethylphthalate	20	18	90	20	100	19	95	95	5.3
Decafluorobiphenyl	20	17	85	16	80	19	95	87	8.8
D_8-Naphthalene	20	19	95	18	90	21	110	98	11
D_{12}-Chrysene	20	14	70	15	75	11	55	67	16

% R = Percent recovery.

TABLE L-68. RECOVERY DATA FOR SELECTED SEMIVOLATILE POHCs FROM SPIKED XAD (FOR MITCHELL SAMPLES)

Compound	μg Added	Rep 1		Rep 2		Rep 3		Average recovery	% RSD
		μg found	% R	μg found	% R	μg found	% R		
Phenol	50	88	180	120	240	180	360	260	35
2,4-Dimethylphenol	20	36	180	27	140	33	170	160	13
Naphthalene	20	80	400	33	170	41	210	160	57
Diethylphthalate	20	28	140	22	110	30	150	130	16
Decafluorobiphenyl	20	28	140	22	110	29	150	130	16
D_8-Naphthalene	20	30	150	24	120	30	150	140	12
D_{12}-Chrysene	20	6.5	33	5.3	27	15	75	45	58

% R = Percent recovery.

TABLE L-69. RECOVERY OF SEMIVOLATILE SURROGATE COMPOUNDS FROM ALL MITCHELL SAMPLES (% RECOVERY)

Sample type	Sample No.	Decafluoro-biphenyl	D_8-Naphthalene	D_{12}-Chrysene
Waste samples				
Aqueous	101	12	84	48
	201	72	64	25
	301	72	84	40
Organic	102	66	80	80
	202	70	85	66
	302	75	115	85
Effluents				
2 XAD + condensate	502	50	80	110
	503	60	50	65
	504	100	110	160
1 Probe rinse + filter	505	Not added	Not added	60
	506	Not added	Not added	110
	507	Not added	Not added	70
Kiln Ash	501	Not added	Not added	70
Blanks				
XAD	211	ND	ND	ND
Filter	508	50	49	95
Spiked water	510	85	95	70
	511	80	90	75
	512	95	110	55
Spiked XAD	507	140	150	33
	508	110	120	27
	509	150	150	75

TABLE L-70. RESULTS OF ANALYSES FOR VOLATILE POHCs IN THREE REPLICATE EXTRACTS OF LIQUID WASTE NO. 202 FROM DU PONT SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Methylene chloride	20,000	19,000	23,000	21,000	10
Chloroform	3,200	2,700	2,700	2,900	10
Carbon tetrachloride	66,000	72,000	66,000	68,000	5.0
Trichloroethylene	3,400	4,900	3,700	4,000	20
Tetrachloroethylene	9,400	13,000	9,900	11,000	18
Toluene	220,000	260,000	240,000	240,000	8.3
cis-1,4-Dichloro-2-butene	15,000	22,000	17,000	18,000	20
3,4-Dichloro-1-butene	17,000	27,000	16,000	20,000	30

TABLE L-71. RECOVERY DATA FOR SELECTED VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF WASTE NO. 202 FROM DU PONT SITE

Compound	Found in[a] unspiked sample μg/g	Amount added μg/g	% Recovery				% RSD
			Spike No. 1	Spike No. 2	Spike No. 3	Average	
Carbon tetrachloride	68,000	68,000	78	96	100	91	13
Trichloroethylene	4,000	8,000	85	81	89	85	4.7
Chloroform	2,900	5,800	180	160	150	160	9.4
Methylene chloride	21,000	42,000	79	80	71	77	6.4
cis-1,4-Dichloro-2-butene	20,000	40,000	0	0	0	0	NA
D_4-1,2-Dichloroethane	-	250	190	66	46	100	77
D_{10}-Ethylbenzene	-	250	130	86	93	100	23

[a] Average of three replicates.

NA = Not applicable.

TABLE L-72. RECOVERIES OF VOLATILE SURROGATE COMPOUNDS FROM DU PONT SAMPLES

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Inflow water	118	100	100
	220	100	94
	318	100	96
Outflow water	119	110	89
	219	90	84
	319	75	92
Waste feeds	102	ND	170
	202-1	ND	110
	202-2	ND	110
	202-3	ND	37
	302	ND	ND
Drum wastes	103	ND	110
	203	90	94
	303	78	89
	104	ND	170
	204	ND	98
	304	ND	94
	105	ND	ND
	205	100	100
	305	120	93
	501	ND	110
	502	ND	74
	503	50	120

ND = Not detected due to small amount of sample analyzed.

TABLE L-73. RECOVERY DATA FOR SELECTED SEMIVOLATILE ANALYTES SPIKED INTO TRIPLICATE SAMPLES OF WASTE SAMPLE NO. 202 FROM DU PONT SITE

Compound	Found in[a] unspiked sample (μg/g)	Added μg/g	% Recovery No. 1	% Recovery No. 2	% Recovery No. 3	% Recovery Average	% RSD
o-Chlorotoluene	180	180	0	0	0	0	0
Phenol	ND	20	500	550	240	430	38
Benzylchloride	3,000	3,000	57	60	43	53	17
Hexachloroethane	560	560	69	61	50	60	16
1,2,3,4-Tetrachlorobutane	3,200	3,200	78	88	47	71	30
Naphthalene	22	20	70	60	20	50	53
p-Chlorophenol	ND	20	0	0	0	0	0
Methylbenzylchloride, all three isomers	790	20	1,300	1,300	1,200	1,300	4

[a] Based on one analysis.

TABLE L-74. RECOVERY DATA FOR SEMIVOLATILE ANALYTES SPIKED INTO TRIPLICATE SAMPLES OF DISTILLED WATER (FOR DU PONT SAMPLES)

Sample Description and No. Compound	Added, µg	Spike No. 1 found	No. 514 % R	Spike No. 2 found	No. 515 % R	Spike No. 3 found	No. 516 % R	Average % R	% RSD
3,4-Dichloro-1-butene	20	7.0	35	5.6	28	4.0	20	28	27
1,4-Dichlorobutane	20	14	70	13	65	12	60	65	7.7
trans-1,4-Dichloro-2-butene	20	13	65	13	65	11	55	62	9.3
o-Chlorotoluene	20	12	60	13	65	9.0	45	57	18
Phenol	20	5.6	28	5.6	28	5.5	28	28	0
Benzyl chloride	20	14	70	14	70	12	60	67	8.6
Hexachloroethane	20	14	70	14	70	12	60	67	8.6
Methylbenzylchloride (all three isomers)	60	37	62	39	65	38	63	63	2.4
1,2,3,4-Tetrachlorobutane	20	13	65	12	60	13	65	63	4.5
Decafluorobiphenyl	20	14	70	14	70	12	60	67	8.6
D_8-Naphthalene	20	15	75	15	75	15	75	75	0
Naphthalene	20	14	70	14	70	14	70	70	0
p-Chlorophenol	20	11	55	11	55	12	60	57	5.1
D_{12}-Chrysene	20	22	110	20	100	20	100	103	5.5

% R = Percent recovery.

TABLE L-75. RECOVERY DATA FOR SEMIVOLATILE ANALYTES SPIKED INTO TRIPLICATE SAMPLES OF XAD RESIN (FOR DU PONT SAMPLES)

Compound	Quantity added µg	% Recovery No. 1	% Recovery No. 2	% Recovery No. 3	% Recovery Average	% RSD
3,4-Dichloro-1-butene	14	59	54	42	52	17
1,4-Dichlorobutane	14	69	71	51	64	17
trans-1,4-Dichloro-2-butene	14	64	61	49	58	14
o-Chlorotoluene	14	79	130	64	91	38
Phenol	14	51	63	36	50	27
Benzylchloride	14	66	61	53	60	11
Hexachloroethane	14	63	56	46	55	16
Methylbenzylchloride, all three isomers	42	43	38	32	38	15
1,2,3,4-Tetrachlorobutane	14	63	58	44	55	18
Naphthalene	34	68	76	53	66	18
p-Chlorophenol	14	41	57	26	41	38
Decafluorobiphenyl	34	62	59	47	56	14
D_8-Naphthalene	34	62	59	47	56	14
D_{12}-Chrysene	34	120	94	150	121	23
C_{13}-Phenol	20	65	60	55	60	8

TABLE L-76. RECOVERY OF SEMIVOLATILE SURROGATE COMPOUNDS FROM DU PONT SAMPLES

Sample type and No.	% Recovery			
	Decafluoro-biphenyl	D_8-naphthalene	D_{12}-chrysene	C^{13}-phenol
Waste samples				
102	140	220	110	NA
202	130	220	130	NA
302	120	230	92	NA
103	150	200	56	NA
203	140	200	64	NA
303	110	170	88	NA
104	180	240	interference	NA
204	180	240	interference	NA
304	150	240	45	NA
105	270	480	40	NA
205	190	340	47	NA
305	110	180	92	NA
501	100	160	35	NA
502	270	400	44	NA
503	120	200	44	NA
MM5 train samples				
508	100	65	70	NA
509	120	100	20	NA
510	110	70	44	NA
130	33	37	150	NA
504	not added	not added	100	NA
505	not added	not added	110	NA
506	not added	not added	75	NA
507	not added	not added	120	NA
Water samples				
118	a	a	a	a
218	44	70	110	NA
318	65	100	90	NA
119	65	100	60	NA
219	110	130	34	NA
319	65	110	70	NA
511	55	75	100	NA
Kiln ash				
117	70	85	75	NA
217	70	80	110	NA
317	65	75	70	NA
513 (blank for kiln ash)	140	130	140	NA

(continued)

TABLE L-76. (continued)

Sample type and No.	% Recovery			
	Decafluoro-biphenyl	D_8-naphthalene	D_{12}-chrysene	C_{13} phenol
Water spikes				
514	70	75	110	NA
515	70	75	100	NA
516	60	75	100	NA
XAD spikes				
5010	62	62	120	65
5011	59	59	94	60
5012	47	47	150	55
Spiked wastes (spiked too low to measure)	not available	not available	not available	NA
	not available	not available	not available	NA
	not available	not available	not available	NA

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[a] Sample extract lost during preparation.

NA = Not applicable because not used.

TABLE L-77. RESULTS OF TRIPLICATE ANALYSES FOR METALS IN PAINT WASTE COMPOSITE SAMPLE NO. 501 FROM DUPONT SITE (μg/g)

Element	Rep 1	Rep 2	Rep 3	Average	% RSD
Al	2,200	1,400	3,900	2,500	51
Al	2,200	1,400	3,900	2,500	51
B	160	140	390	230	60
Ba	5.6	4.9	10	6.8	40
Ca	< 140	< 140	150	NA	NA
Cu	160	110	310	190	54
Fe	840	800	1,500	1,000	38
Fe	890	840	1,600	1,100	38
Ni	< 61	82	< 61	NA	NA
P	57	< 49	98	NA	NA
Sb	< 12	151	23	NA	NA
Sn	18	< 12	25	NA	NA
Ti	38	1,100	170	440	130
Zn	< 12	23	19	NA	NA

TABLE L-78. RECOVERY DATA FOR METALS SPIKED INTO TRIPLICATE SAMPLES OF PAINT WASTE COMPOSITE NO. 501 FROM DU PONT SITE (μg/g)

Element	Unspiked	Spike level	Rep 1	% Recovery	Rep 2	% Recovery	Rep 3	% Recovery	Average % recovery	% RSD
As	< 24	380	280	74	380	100	380	100	91	16
B	160	360	490	92	730	160	780	170	140	30
Ba	5.6	370	280	74	380	100	390	100	91	16
Be	< .18	360	250	69	340	94	350	97	87	18
Ca	< 140	340	300	88	420	120	420	120	110	17
Cd	< 5.0	360	250	69	350	97	360	100	88	19
Co	< 3.0	360	260	72	360	100	360	100	90	18
Cr	< 5.2	370	270	73	370	100	380	100	91	17
Hg	< 22	370	270	73	360	97	370	100	90	16
Mg	< 16	350	270	77	360	100	360	100	92	14
Mn	< 16	360	270	75	360	100	370	100	92	16
Mo	< 3.2	360	260	72	360	100	360	100	90	18
Ni	< 61	360	270	75	370	100	380	110	95	19
P	57	350	290	67	430	110	430	110	96	26
Pb	< 19	360	270	75	370	100	380	110	95	19
Sb	< 12	360	250	69	350	97	350	97	88	18
Sn	18	360	270	70	370	98	380	100	89	19
Ti	38	370	330	79	500	120	460	110	100	21
Tl	< 2.3	370	260	70	350	95	360	97	87	17
Y	< 2.3	360	260	72	350	97	360	100	90	17
Zn	22	360	260	66	360	94	360	94	85	19

TABLE L-79. RESULTS OF TRIPLICATE ANALYSES FOR VOLATILE POHCs IN LIQUID ORGANIC WASTE NO. 301-1 FROM TWI SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Methylene chloride	680	150	180	340	88
Chloroform	ND	83	88	86	NA
1,1,1-Trichloroethane	ND	230	470	350	NA
Carbon tetrachloride	9,900	9,700	10,000	9,900	1.5
Trichloroethylene	10,000	13,000	11,000	11,000	13
Benzene	74,000	41,000^s	44,000^s	53,000	34
Tetrachloroethylene	ND	260	180	220	NA
Toluene	380,000^s	T	T	NA	NA
Chlorobenzene	530	510	460	500	7.2

s = Ion used was saturated.

ND = Not detected.

NA = Not applicable.

T = Peak truncated due to high concentration.

TABLE L-80. RESULTS OF REPLICATE ANALYSES FOR VOLATILE POHCs IN LIQUID AQUEOUS WASTE NO. 308-1 FROM TWI SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Methylene chloride	49	76	56	60	2.3
Bromochloromethane	22	22	19	21	8
Chloroform	3.5	4.5	1.2	3.1	55
Methylene bromide	4,700	4,900	4,500	4,700	4
Benzene	6.3	8.2	2.6	5.7	50
Tetrachloroethylene	1.2	1.1	ND	1.2	NA
Toluene	19	23	19	20	11

ND = Not detected.

TABLE L-81. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF ORGANIC WASTE NO. 301 FROM TWI SITE

Compound	Found in unspiked sample (μg/g)	Added (μg/g)	% Recovery				% RSD
			Spike no. 1	Spike no. 2	Spike no. 3	Average	
Carbon tetrachloride	9,870	19,700	66	81	76	74	10
Trichloroethylene	11,300	22,600	69	69	69	69	0
Chloroform	86	170	120	150	76	120	32
Methylene bromide	ND(a)	1,000	160	110	140	140	18
Chlorobenzene	500	1,000	100	90	90	93	6.2
D_4-1,2-Dichloroethane	-	250	ND(2)	ND(2)	ND(2)	ND(2)	NA
D_{10}-Ethylbenzene	-	250	98	86	97	94	7.1

ND(a) = Not detected in unspiked sample.

ND(2) = Not detected in spiked sample due to small amount of sample analyzed.

NA = Not applicable.

TABLE L-82. RECOVERY DATA FOR VOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF AQUEOUS WASTE NO. 308 FROM TWI SITE

Compound	Found in unspiked sample (μg/g)	Added (μg/g)	% Recovery Spike No. 1	% Recovery Spike No. 2	% Recovery Spike No. 3	% Recovery Average	% RSD
Carbon tetrachloride	NA	1,000	14	12	7.5	11	30
Trichloroethylene	NA	1,000	37	35	29	34	12
Chloroform	3.1	1,000	20	24	21	22	9.6
Methylene bromide	4,700	9,400	56	63	63	61	6.7
Tetrachloroethylene	1.2	1,000	53	56	54	54	2.8
D_4-1,2-Dichloroethane	-	250	23	8	14	15	50
D_{10}-Ethylbenzene	-	250	39	38	33	37	8.8

NA = Not applicable.

TABLE L-83. RECOVERIES OF VOLATILE SURROGATE COMPOUNDS FROM TWI SAMPLES

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Well water	112	100	100
	212	110	100
City water	113	97	96
	213	81	91
	313	87	92
	413	83	100
Recirculated water	114	81	80
	214	88	94
	314	110	99
	414	91	93
Return water	115	99	95
	215	79	98
	315	100	120
	415	110	94
Organic waste	101	71	99
	201	52	96
	301-1	ND	320
	301-2	67	110
	301-3	120	78
	401	ND	89
	460	61	82
	601	95	110
	701	68	130
	801-1	90	110
	801-3	91	260
Aqueous waste	108	97	91
	208	96	130
	308-1	98	140
	308-2	110	110
	308-3	92	100
	408	89	86
	461	110	110
Solid waste composite	110	73	130
	210	84	140
	310	120	I.T.
	410	ND	ND
	462	ND	ND
	810	110	110

(continued)

TABLE L-83. (continued)

Sample type	Sample No.	D_4-1,2-Dichloroethane	D_{10}-Ethylbenzene
Chamber ash	111	92	67
	211	98	72
	311	81	60
	411	99	110
Fuel oil	209	130	71
Water tank solids	316	100	100

ND = Not detected due to small amount of sample analyzed.

I.T. = Co-eluting interference peak truncated and prevented quantification of d_{10}-Ethylbenzene.

TABLE L-84. RESULTS OF ANALYSES FOR SEMIVOLATILE POHCs IN REPLICATE EXTRACTS OF ORGANIC WASTE SAMPLE NO. 301 FROM TWI SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Naphthalene	40	24	36	33	25
Di-n-butyl phthalate	130	160	130	140	12
Bis(2-ethylhexyl)phthalate	240	220	220	230	5

TABLE L-85. RESULTS OF ANALYSES FOR SEMIVOLATILE POHCs IN REPLICATE EXTRACTS OF SOLID WASTE COMPOSITE NO. 410 FROM TWI SITE (μg/g)

Compound	Rep 1	Rep 2	Rep 3	Average	% RSD
Naphthalene	8,100	11,000	11,000	10,000	17
Hexachlorobutadiene	320	480	460	420	21
Hexachlorocyclopentadiene	16,000	20,000	18,000	18,000	11
Octachlorocyclopentene	390	490	460	450	11
Hexachlorobenzene	< 30	< 30	< 30	< 30	0
Di-n-butyl phthalate	120	< 30	< 30	56	100
Bis(2-ethylhexyl)phthalate	< 30	< 30	< 30	< 30	0

TABLE L-86. RECOVERY DATA FOR SELECTED SEMIVOLATILE POHCs SPIKED INTO TRIPLICATE SAMPLES OF SOLID WASTE COMPOSITE NO. 410 FROM TWI SITE

Compound	Found in[a] unspiked sample µg/g	Amount added µg/g	% Recovery				% RSD
			Spike no. 1	Spike no. 2	Spike no. 3	Average	
Decafluorobiphenyl	-	250	170	210	220	200	13
1,2,4-Trichlorobenzene	ND	100	170	180	220	190	14
Naphthalene	10,000	10,000	30	60	80	56	44
D_8-naphthalene	-	250	160	190	200	180	11
Hexachlorobutadiene	420	840	140	200	220	190	22
Hexachlorocyclopentadiene	18,000	18,000	11	39	61	37	68
Octachlorocyclopentene	450	900	150	180	190	170	12
Hexachlorobenzene	15	30	160	180	220	190	16
Di-n-butyl phthalate	56	110	76	49	58	61	22
Chlordane	ND	2,000	55	55	48	53	8
D_{12}-chrysene	-	250	44	44	40	43	5
Bis(2-ethylhexyl)phthalate	4.8	100	57	57	53	56	4

[a] Average of three replicates.

TABLE L-87. RECOVERY DATA FOR SEMIVOLATILE ANALYTES SPIKED INTO TRIPLICATE SAMPLES OF XAD RESIN (FOR TWI SAMPLES) (μg/g)

Sample no.		Spike no. 1		Spike no. 2		Spike no. 3			
		5010		5011		5012			
Compound	Added	Found	% R	Found	% R	Found	% R	Average % R	% RSD
Decafluorobiphenyl	34	30	88	27	79	21	62	76	17
1,2,4-Trichlorobenzene	20	19	100	18	90	14	70	87	18
Naphthalene	34	36	110	38	110	26	76	99	28
D_8-naphthalene	34	31	91	28	82	23	68	80	14
Hexachlorobutadiene	20	20	100	18	90	14	70	90	18
Hexachlorocyclopentadiene	20	5.0	30	11	60	8.1	41	43	35
Octachlorocyclopentene	20	18	90	19	100	16	80	90	11
Hexachlorobenzene	20	21	110	20	100	18	90	100	10
Di-*n*-butyl phthalate	20	17	90	19	100	21	110	100	10
Chlordane	100	69	69	65	65	91	91	75	19
D_{12}-chrysene	34	24	71	18	53	28	82	69	21
Bis(2-ethylhexyl)phthalate	20	19	100	16	80	23	120	100	20

% R = Percent recovery.

TABLE L-88. RECOVERIES OF SEMIVOLATILE SURROGATE COMPOUNDS FROM TWI SAMPLES (% RECOVERY)

Sample type and no.	Decafluorobiphenyl	D_8-naphthalene	D_{12}-chrysene
Waste samples			
101	56	60	15
201	72	68	17
301-1B-1	92	96	20
301-1B-2	56	60	20
301-1B-3	84	88	20
401	84	80	22
460	64	60	16
110	48	48	33
210	48	48	44
310	48	48	35
410-1B-1	68	68	28
410-1B-2	200	180	36
410-1B-3	190	180	27
462	110	100	56
MM5 train samples			
5001	110	100	36
5002	90	90	42
5003	80	90	34
5004	100	110	41
5005	Not added	Not added	90
5006	Not added	Not added	95
5007	Not added	Not added	90
5008	Not added	Not added	100
5005 (5014)	85	90	95
5009	60	70	65
Chamber ash			
111	210	220	20
211	120	140	14
311	150	160	24
411	190	120	15
Water tank solids			
316	60	110	10
Spiked XAD			
5010 no. 1	88	91	71
5011 no. 2	79	82	53
5012 no. 3	62	68	82
Spiked waste			
410 no. 1	170	160	44
410 no. 2	210	190	44
410 no. 3	220	200	40

TABLE L-89. RESULTS OF REPLICATE ANALYSES FOR METALS IN ORGANIC WASTE NO. 401 FROM TWI SITE (μg/g)

Element	Rep 1	Rep 2	Average	% RSD
Al	1,700	1,700	1,700	0
Al	1,800	1,700	1,800	4.0
B	22	25	24	9.0
Ba	1,200	1,200	1,200	0
Be	0.32	0.32	0.32	0
Ca	2,800	2,900	2,900	2.5
Cd	61	60	61	1.2
Co	6.0	6.4	6.2	4.6
Cr	310	320	320	2.2
Cu	100	99	100	0.7
Fe	1,000	1,300	1,200	18
Fe	1,000	1,300	1,200	18
K	140	100	120	24
Mg	340	340	340	0
Mn	13	16	15	15
Mo	32	33	33	2.2
Na	440	440	440	0
P	180	190	190	3.8
Pb	1,500	1,500	1,500	0
Sb	< 24	28	NA	NA
Se	< 160	180	NA	NA
Sn	< 14	24	NA	NA
Ti	26	26	26	0
Zn	2,200	2,200	2,200	0

TABLE L-90. RESULTS OF DUPLICATE ANALYSES FOR METALS IN SOLID WASTE COMPOSITE NO. 410 FROM TWI SITE (μg/g)

Element	Rep 1	Rep 2	Average	% RSD
Al	1,100	1,400	1,300	17
Al	1,100	1,300	1,200	12
B	42	30	36	24
Ba	23	32	28	23
Be	0.96	1.4	1.2	26
Ca	2,400	3,600	3,000	28
Cr	13	18	16	23
Cu	34	57	46	36
Fe	9,400	13,000	11,000	23
Fe	9,400	13,000	11,000	23
K	360	320	340	8.3
Mg	520	710	620	22
Mn	52	60	56	10
Na	160	120	140	20
Ni	9.1	11	10	13
P	54	71	63	19
Pb	49	75	62	30
Ti	83	100	92	13
Y	1.6	2.5	2.1	31
Zn	56	69	63	15

TABLE L-91. RECOVERY DATA FOR METALS SPIKED INTO DUPLICATE SAMPLES OF ORGANIC WASTE SAMPLE NO. 401 FROM TWI SITE (μg/g)

Element	Unspiked	Spike level	Spiked replicate no. 1	% Recovery	Spiked replicate no. 2	% Recovery	Average % recovery
As	< 23	42	60	140	54	130	140
B	22	40	61	98	60	95	97
Be	< 0.32	41	46	110	46	110	110
Cd	61	40	110	120	110	120	120
Co	< 6.0	39	49	130	48	120	130
Cu	100	39	160	150	160	150	150
Hg	< 50	41	51	120	61	150	140
Mn	13	40	60	120	60	120	120
Mo	32	40	77	110	78	110	110
Ni	< 4.1	40	36	90	34	90	90
P	180	39	220	100	240	150	130
Sb	< 24	40	48	120	62	160	140
Sn	< 14	40	55	140	62	160	150
Ti	26	41	70	110	71	110	110
Tl	< 22	41	64	160	56	140	150
Y	< 0.35	40	45	110	44	110	110

TABLE L-92. RECOVERY DATA FOR METALS SPIKED INTO DUPLICATE SAMPLES OF SOLID WASTE COMPOSITE NO. 410 FROM TWI SITE (μg/g)

Element	Unspiked	Spike level	Spiked replicate no. 1	% Recovery	Spiked replicate no. 2	% Recovery	Average % recovery
As	< 23	42	57	140	67	160	150
B	42	40	97	140	94	130	140
Be	0.96	41	45	110	45	110	110
Cd	< 2.0	40	46	120	46	120	120
Co	< 6.0	39	48	120	48	120	120
Cu	34	39	94	150	97	160	150
Hg	< 50	41	70	170	71	170	170
Mn	52	40	100	120	110	150	130
Mo	< 3.5	40	45	110	45	110	110
Ni	9.1	40	52	110	54	110	110
P	54	39	95	110	88	87	96
Sb	< 24	40	34	85	44	110	98
Sn	< 14	40	50	130	50	130	130
Ti	83	41	140	140	120	90	120
Tl	< 22	41	34	83	36	87	85
Y	1.6	40	44	110	44	110	110